

**DEVELOPMENT OF STABLE BENTONITE FOR  
DRILLING FLUID FORMULATIONS USING LOCAL  
SOURCES**

BY

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*I dedicate this to my mother and father*

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In the name of ALLAH, the Most Beneficent, the Most Merciful.

﴿ And Allah will raise up in ranks those who believed among you and those who have been given knowledge, Allah is aware of what you do ﴾ Surat Al-Mujadila(Ch.58-11)

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## LIST OF ABBREVIATIONS

A-1	:	Bentonite-1 Dry sieved through 200 mesh (size < 38 $\mu\text{m}$ ).
API	:	American petroleum institute.
B-3	:	Bentonite-2 dry sieving through 200 mesh (size < 38 $\mu\text{m}$ ).
B-7	:	Bentonite-2 raw sample.
Bent-1	:	Local bentonite batch-1.
Bent-2	:	Local bentonite batch-2.
BN-6	:	Bentonite-1 Purified using Hexameta phosphate and sedimentation.
BN-7	:	Bentonite-1 Purified using Hexameta phosphate and sedimentation.
HPHT	:	High pressure and high temperature condition.
OCMA	:	Oil company materials association.
PSA	:	Particles size analysis.
R-1	:	Bentonite-1 raw sample.
S-1	:	Bentonite-1 purified using wet sieving, size < 38 $\mu\text{m}$ .
S-2	:	Bentonite-1 purified using wet sieving, 38 $\mu\text{m}$ < size < 64 $\mu\text{m}$ .
S-3	:	Bentonite-1 purified using wet sieving, 63 $\mu\text{m}$ < size < 75 $\mu\text{m}$ .
S-4	:	Bentonite-1 purified by wet sieving, size > 75 $\mu\text{m}$ .
SED-1	:	Bentonite-1 purified using sedimentation from supreme solution.
W-1	:	Bentonite-1 Purified by wet sieving, 200 mesh (size < 38 $\mu\text{m}$ ).



## **ABSTRACT**

Full Name : Musaab Ibrahim Magzoub Elhag  
Thesis Title : Development of Stable Bentonite for Drilling Fluid Formulations  
Using Local Sources  
Major Field : Petroleum Engineering  
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Drilling fluid is the key factor in drilling operation and accessing the oil and gas reservoirs. The future trends now are heading for a new development and alternatives to cut the expenses of drilling operation as low as possible. Bentonite is used as a viscofier, fluid loss control additive, and as a weighting material in water-based drilling fluids. The type of bentonite used in drilling fluid formulation is sodium bentonite which has high dispersion properties and high swelling capacity. Saudi Arabia has a huge bentonite clay deposit resources. This enormous amount of bentonite can be evaluated and enhanced in order to be used as drilling fluid and cementing additive. Bentonite natural resources from Khulays Area in North Jeddah, Saudi Arabia, was investigated and found to be calcium bentonite which is not suitable for drilling fluids, because it has low swelling capacity and bad rheological properties.

In this study the local bentonite was fully characterized, chemical composition and physical properties were identified. A pure bentonite was extracted through simple designed purification process. Local bentonite was sodium activated using a new proposed method of thermo-chemical upgrading process with soda ash, which proved to add a crucial affect in the activation process. The experimental work included

measuring the rheological properties at low and HPHT of drilling fluid, suspension stability of bentonite, swelling, and filtration.

The results obtained from this study showed that the properties of treated local bentonite developed through thermo-chemical upgrading, outperformed the commercial standard bentonite. The rheological properties enhanced and achieved the target of standard API specifications. Therefore, the obtained pure Ca-bentonite from natural deposit in Khulays area can be modified to Na-bentonite and sufficiently used in drilling fluid and cementing additives using thermal upgrading method.

: مصعب إبراهيم مجذوب الحاج

: تطوير بنتونايت مستقر للاستخدام في تركيبات موانع الحفر من مصادر محلية

: هندسة بترول

تاريخ الدرجة العلمية: 1436

مائع الحفر هو العامل الأساسي في عملية الحفر للوصول إلى مكامن ال . الاتجاهات المستقبلية الآن تتجه لتطوير بدائل جديدة لخفض نفقات عملية الحفر عند أدنى مستوى ممكن. يستخدم البنتونيت لزيادة لزوجة موانع الحفر و تقليل فقد الماء، وكما مادة لزيادة كثافة ووزن سوائل الحفر التي تستخدم الماء لتحضير السؤال WBM. البنتونيت المستخدم في الحفر هو بنتونايت الصوديوم Na-bentonite الذي له خصائص تشتت عالية في الماء وقدرة عالية الانتفاخ. المملكة العربية السعودية لديها موارد ضخمة من صلصال البنتونيت. هذه الثروة الطبيعية الهائلة من البنتونيت يمكن تقييمها وإستصلاحها من أجل استخدامها في موانع الحفر كبديل للبنتونايت المضاف. الموارد الطبيعية للبنتونيت من منطقة خليص في شمال جدة، في المملكة العربية السعودية، تم التحقيق عنها ووجدت انها تحتوي علي بنتونيت من نوع الكالسيوم Ca-bentonite. هذا النوع لا يناسب إستخدامات موانع الحفر، الانتفاخ منخفضة وخصائص ريولوجية سيئة.

في هذه الدراسة تمت دراسة البنتونيت من المصادر المحلية في المملكة السعودية، تم دراسة التركيب الكيميائي والخصائص الفيزيائية. تم إستخلاص البنتونيت النقي من خلال عملية تنقية بسيطة تم تصميمها للحصول علي اعلي . تنشيط البنتونايت المحلي عن طريق تنشيط الصوديوم بإضافة كربونات الصوديوم التي يطلع عليها مسمي رماد الصوديوم Soda Ash, وذلك بإستخدام طريقة جديدة مبتكرة لتفعيل عملية التنشيط. الطريقة الجديدة المقترحة لتنشيط الصوديوم في البنتونايت المحلي تستخدم معالجة حرارية وكيميائية مع إضافة رماد الصودا، هذه المعالجة ثبت أنها تؤثر تأثيرا أساسيا و حاسما في عملية التنشيط. التجارب المعملية شملت علي قياس خصائص الانسيابية في درجة حرارة الغرفة وفي درجات الحرارة العالية لتحقيق الثبات الحراري.

الاستقرار وتعليق البنتونيت في الماء، وخاصة الإنتفاخ، وخاصة ترشيح وفقد الماء عند إستخدام البنتونايت في تركيب موانع الحفر.

أظهرت النتائج المتحصل عليها من هذه الدراسة أن خصائص البنتونيت المحلي الذي تم تطويره من خلال المعالجات الحرارية والكيميائية، فاق أداء البنتونيت القياسي التجاري. خواص الريولوجية تعززت وحققت الدرجة المطلوبة وفقا لمواصفات القياسية المنصوص عليها عالميا. بالتالي فإن بنتونايت الكالسيوم النقي الذي تم إستخلاصه من المصادر الطبيعي المحلية في منطقة خليص يمكن تنشيطها وتحويلا لنوع بنتونايت الصوديوم. يمكن لهذا البنتونايت ان يستخدم بشكل فعال كمادة مضافة في موانع الحفر بعد معالجته باستخدام رماد الصودا عن طريقة التطوير الحراري الكيميائي.

# **CHAPTER 1**

## **INTRODUCTION**

Drilling a well from surface to target zones is the most expensive part in the oil industry, and drilling fluids share almost 50% of drilling cost. Bentonite is used in water based Mud to formulate the body of drilling fluids, works as viscofier additives and reduce water losses.

Physical and chemical properties of drilling fluid depend on the type of chemical additives added which have different behaviors and physical characteristics <sup>[1]</sup>. The best combination of these additives will provide the required functions of the drilling fluid such as; carrying the cuttings from downhole to the surface, cooling and lubricating the drilling bit, reducing friction and drag, maintaining wellbore stability, preventing influx of fluids from wellbore, and forming thin and low permeable filter cake to control water losses and support hole wall and also not cause damage the production zone <sup>[2]</sup>.

Drilling through different formation sections put drilling fluids in a great challenge to select the suitable formulation to cope with certain situations, like drilling through shale formation, salt domes, deep water drilling and high pressure high temperature (HPHT) reservoirs. Water based drilling fluid is less expensive than oil based mud and can drill most of the well sections specially the main section where formation damage is not critical, and if WBM is properly formulated, its application can be extended. Certain chemicals are added to enhance mud filtration and other properties. Additives such as lost circulation

materials (LCM), inhibitors, weighing material, and viscofier, while bentonite can be up to 80% in WBM.

Raw bentonite from natural resources is purified from impurities and treated to upgrade its properties in order to be used in drilling mud. API and OCMA specification are the guide line for the evaluation of bentonite performance, from different locations worldwide, raw bentonite has been a potential research area, and Saudi Arabia has a huge bentonite clay deposit resources.

Khulays area in Saudi Arabia, 95 Km north of Jeddah has the largest clay deposit in the kingdom, 800m long  $\times$  600 m wide, the thickness is about 200-300 m, estimated to contain 3 million tons of raw bentonite clay, 90% smectite with 10% kaolinite and/or 5-15% quartz (Mineral Resources in Saudi Arabia, ministry of petroleum 1995 CH6). It's known to be calcium bentonite (Ca-bentonite).

Calcium bentonite has lower swelling capacity compared to sodium bentonite, the best quality bentonite can produce organo clays or nanoclays used in polymers, paints, medicine and other industrial applications <sup>[3]</sup>. For drilling mud sodium exchange bentonite is preferable and can be obtained by upgrading the ca-bentonite by treatment with soda ash to increase sodium to calcium ratio and enhance the swelling characteristics <sup>[4]</sup>. Even high grade bentonite like Khulays deposits (over 90 % montmorillonite) still need some treatments to match drilling requirements, only 2-4 % of soda ash ( $\text{Na}_2\text{CO}_3$ ) can greatly improve the bentonite quality (Foster, 1953), also sufficient Mg exchange ions are important to provide good gel strength. <sup>[5]</sup>

Analysis such as XRD and XRF is used to determine the mineralogy composition and types of component in raw deposits, drilling fluids laboratories can simulate the downhole condition to evaluate filtration properties, mud cake thickness, mud rheological properties, and thermal stability based on API specifications.

Local raw bentonite purification and beneficiation should be achieved using cheap and simple process. Mechanical methods by sieving or chemical methods by adding materials to bentonite solution to precipitate the impurities can be used. Stability of pure bentonite can be evaluated using zeta potential (ZP) concept which is a measurement of particles charges, if ZP is above +20 or below -20 the solution is said to be stable. **Fig.1** shows some typical zeta potential for bentonite in range of different PH.<sup>[6]</sup>

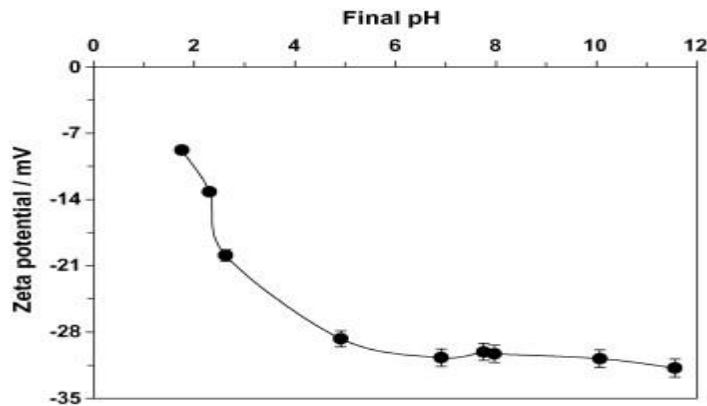


Figure 1 Zeta Potential vs PH of Bentonite

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Types of Drilling Fluid**

There are several different types of drilling fluids suitable for different specific conditions. Drilling fluid bases such as water based WBM, oil based OBM, gas based GBM or synthetic based are used in different situations with wide range of chemical additives <sup>[1]</sup>. Water based drilling fluids are the most preferred type <sup>[7]</sup>. Many factors are considered in selection of drilling fluid base and chemical additives. Safety and environment are the most important ones; drilling fluid should provide the primary control over pressure and be environmentally friendly <sup>[1]</sup>. Other factors are high temperature, high pressure performance, loss zones and expected shale related problems.

Selection and designing of proper drilling fluid should provide the essential functions that are important for successful drilling. A fluid is required in the wellbore for cleaning the hole and transporting all the cutting to the surface and applying sufficient hydrostatic pressure to balance formation pressure. Circulating of drilling fluids act as a cooling and lubricating material, ensure well stability by supporting the hole before casing being set, and avoid formation damage <sup>[8]</sup>.

##### **2.1.1 Air Drilling**

According to Leonard W.Cooper(1977), air and gas were introduced as drilling fluids in 1950.As world demand for oil increased; the drilling main objective was to achieve high



penetration rates, and low cost fluid to remove drilling-cuttings to the surface. Air provide significant high rate of penetration ROP and longer bit life <sup>[9]</sup>.

Air technology was developed through time and had many successful deployments. K.Nicolson (1953) reported using air as circulating fluid to drill many wells in California, US. In addition to the low cost of air drilling, this technique also used as alternative method when sever loss zone is encounter or water source is difficult to be supplied. Operators in that filed drilled 14 wells with Air, and rate of penetration ROP for these well was higher that wells drilled using mud by 40 to more than 200 % <sup>[10]</sup>.

#### Ideal Candidate for Air Drilling <sup>[8]</sup>

- Long wellbore sections with minimal formation pressures
- Medium - hard formations w/ low ROP
- Deviation problems due to high formation dip angles
- Lost circulation problems
- Formations that have minimum water influx
- Water sensitive formations (shales) are time sensitive to sloughing.
- Hydrocarbon zones are typically gas bearing.

The drawback of Air drilling is related to the mechanisms of this method, additional equipment is needed. Teddy Gilbert (1968) highlighted some limitations, such as large compressors that are needed to supply sufficient air to the system. High annular velocity (3000 fpm) is needed to ensure hole cleaned properly. Weak formation can be unstable

and cause hole enlargement, also air requires the hole to be dry without any water influx [11].

### **2.1.2 Foamable Drilling Fluid**

Foam is a mixture of air with water and surfactant, introduced to handle water that cannot be carried to the surface with air, foamable drilling fluids can cope with difficulties in offshore drilling. Deep water causes high pressure due to the weight of mud inside the lengthy large riser, lighter fluid is needed. Patty et al (1998) proposed a foamable drilling fluid in a formula of prehydrated bentonite with a group of other chemicals such as alcohol sulfates, salts, and others. They succeeded in stabilizing the foam to be used as drilling fluid which solved fluid loss problems, reduced mud cake permeability and improved hole cleaning by increasing mud carrying capacity [12].

Drilling with foam may complicate the hydraulic design of drilling fluid, because it deals with compressible fluid instead of incompressible, which makes it difficult to estimate the pressure change and hydraulics inside the hole by the conventional calculation methods and simulators are needed to conduct the numerical [13].

Paknejad et al. (2009) did a sensitivity study on the key factors that affecting performance of foam drilling. They showed that the performance of foam drilling was greatly affected by cutting size and concentration of drilling-cuttings and injection rate of foam is depending on the depth [14].

Lage et al.(1996) reported many cases in Brazil where foam was used as drilling fluid, firstly it was used in “Carmopolis” depleted field, and the field was experiencing total fluid loss in some drilling sections. Foam drilling was recommended as the best way to

overcome this total loss problem, fluid losses were around 10,000 bbls. This huge loss of fluid and also cuttings into the formation caused severe formation damage, in some cases well cannot back to produce normally until 6 months. Using foam saved time and cost and eliminated the formation damage <sup>[15]</sup>.

### **2.1.3 Mist Drilling**

Mist means air drilling with the addition of liquids usually water, soap and chemical inhibitors. Mixture of water and soap is added to the air stream at surface at a controlled rate to improve annular hole cleaning. Different media can be used in the misting process such as water, surfactants, etc.

During misting the annular pressure increases so the ROP will drop versus dusting applications (Additional Air Volume can help improve ROP) <sup>[8]</sup>.

Misting is used when wellbore becomes wet due to fluid influx, annular cleaning problems lead to inconsistent flow at the blooie line (high pressure). Wellbore fluid influx is up to 100+ gpm / 150 Bbls/Hr but is dependent upon air volume, reservoir produces large amount of gas condensate which creates hole cleaning problem, or hole showing fill after connections suggesting (sloughing problems).

### **2.1.4 Gaseated or Aerated Drilling**

Air or Nitrogen is added to the liquid phase of the drilling fluid, this will lower the effective mud weight, and this can be accomplished by injecting air/nitrogen directly into the standpipe. This can also be achieved using parasite string or concentric casing strings <sup>[8]</sup>.

## **2.2 Bentonite**

### **2.2.1 Bentonite in Drilling Fluids**

Bentonite is used in drilling fluids to increase viscosity and reduce filtration losses. When bentonite interacts with water it swells because it is hydrophilic and adsorbs water. Good quality bentonite when used in drilling fluids will give the required viscosity and acceptable filtration loss <sup>[4, 5, 8,16]</sup>.

Earl V.Shannon et al.(1926) described the origin of bentonite rocks as metamorphic rocks derived from volcanic glassy ash, composed of Montmorillonite minerals <sup>[17]</sup>.

The volcanoclastic or pyroclastic rocks are composed mainly from Montmorillonite from the Smectite group <sup>[4, 16,18]</sup>.

Generally three main of types of bentonite <sup>[19]</sup>:

- (1) Sodium montmorillonite which is naturally activated sodium bentonite
- (2) Calcium montmorillonite which naturally activated calcium bentonite
- (3) Artificial Sodium activated bentonite

There is also some other ions activated bentonite such as potassium-bentonite, magnesium-bentonite, and lithium-bentonite. Naturally Na-bentonite has the ability to absorb water and increase viscosity, and form thixotropic suspensions. Ca-bentonite has low swelling capability and form unstable suspension.

Bentonite Quality can be determined by the content of materials other than Montmorillonite, the presence or absence of small amounts of extending polymers and the size and charge of the Montmorillonite platelets <sup>[20]</sup>.

OCMA specified OCMA grade bentonite to set a standard for that used in drilling fluids. They defined drilling grade bentonite as clay mineral smectite, which prepared and chemically treated to meet OCMA specifications shown in the following <sup>[21]</sup>**Table.1**

[API 13A Specification for drilling fluid materials, 15<sup>th</sup> ED 1993]

**Table 1 API 13A Specification for Drilling Grade Bentonite**

<b>Property</b>	<b>Specified Limits.</b>
Viscometer dial Reading at 600 rpm	Minimum 30
Yield Point to Plastic viscosity Ratio	Maximum 3
Filtration volume at 30 min	Maximum 15 cm <sup>3</sup>
Residue after sieved with 200 mesh (75 µm)	Maximum 4.0 % by weight > 75 µm
Moisture	Maximum 10.0 % by weight

Non-treated bentonite specified by OCMA is not chemically-treated clay, and only dried out of moisture and grinded. Composed from smectite minerals, should meet the physical specifications such as yield to plastic viscosity ratio and plastic viscosity showed in the following <sup>[21]</sup>**Table.2**

**Table 2 OCMA Non Treated Bentonite Specifications**

<b>Property</b>	<b>Specified limits</b>
Yield Point to Plastic viscosity Ratio	Maximum 1.5
Plastic viscosity	Maximum 10 cP
Filtrate volume	Maximum 12.5 cm <sup>3</sup>

### **2.2.2 Raw Bentonite Beneficiation**

Raw bentonite exists in nature associated with many minerals impurities, with different composition due to different locations and environment, elements other than Montmorillonite should be removed <sup>[22]</sup>.

Pure bentonite can be obtained by dispersing the clay into deionized water (A. S. Ahmed et al.(2012)). The method is based on Stoke's law to calculate retention time of sedimentation. Raw bentonite was put into water and was allowed to swell for 24 hours, then stirred for 30 min. before it settled in a container for a period of time that recommended by Stoke's law (10 hrs.).

After retention time the final solution will be containing the particles sizes less than 2  $\mu\text{m}$  as the size of Montmorillonite particles, which is collected and dried at 90°C, grounded and sieved through 200 meshes <sup>[3]</sup>.

Other methods to purify raw bentonite are the dry method and wet method described by O. JAMES et al.(2008). In dry method no water is used, raw clay is crushed to small

pieces, dirt is removed and big impurities are removed with 2mm sieve. bentonite obtained by sieving from grinded dry samples after removing moisture at 150 °C-200°C.

Wet method uses a Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), which has the ability to remove any organic and inorganic matter in the clay. Raw clay is put in water with only 10%  $\text{H}_2\text{O}_2$  solution, suspension is allowed to stand for a while depend on the amount of clay (100 gm in 1 L water wait 2 hrs.). Supreme water collected and poured into sodium hydroxide solution of 0.5 M concentration and settled for 24 hrs., then diluted with deionized water and siphoned off the <2 micron clay, the operation of diluting with water and siphoning is repeated until very little residue of clay

The Disodium Edetate ( $\text{Na}_2\text{EDTA}$ ) can be used to purify raw bentonite, a method of Isaac (1969), where crushed and a screened raw bentonite is dispersed in 0.01 M  $\text{Na}_2\text{EDTA}$  in 0.1 M NaCl, Stirred for 30 minutes and the suspension of solution put in shaker for 30 minutes, then allowed to stand for 48 hours, this will give pure clay particles of size 2  $\mu\text{m}$  which can be extracted through repeated sedimentation process followed by siphoning <sup>[23]</sup>.

Purification of bentonite is the process of removing all impurities rather than Montmorillonite, including clay and non-clay impurities. Paul Schick (1975) invented a way to purify raw bentonite using sodium polymeta Phosphate ( $\text{NaPO}_3$ )<sub>n</sub>. he succeeded in extracting pure bentonite with high quality even good for food industry applications. All impurities were removed such as quartz, feldspar and illite. The purification was achieved through mixing of aqueous slurry of clay sample with aqueous dilute solution of sodium

hexametaphosphate, this causes the impurities to settle down and separate from the solution.

Using sedimentation with distilled water is not an efficient way for purification of raw bentonite. Because it consumes lots of water and time, while using as sodium hexametaphosphate with the solution increased sedimentation rate and saved time. Almost 90% of impurities removed and analysis for the settled precipitates showed that it contains most of the impurities. Sweet water was used to prepare the clay slurry, bentonite to water ration between 1:1.5 and 1:2. Very little sodium polymetaphosphate was used as 0.5%. Solution agitated 3 to 5 minutes, later 5 to 10 minutes was enough to precipitate all material rather than Montmorillonite. Then bentonite can be obtained from suspension by centrifuging or any other mean <sup>[22]</sup>.

### **2.2.3 Beneficiation and Evaluations of Local Bentonite**

Local bentonite from Nijeria raw clay in Gombestate was examined by A.S.Ahmed et al.(2012), mineralogy and chemical composition was investigated. Bentonite beneficiation carried out by crushing clay sample to coarse powder and soaks it in water with stirring for 3 hours, then left for 25 hrs, for sedimentation process to take place. Colloid bentonite was sieved through a 230 mesh (63  $\mu\text{m}$ ).

Raw bentonite treated by calcination and acidification, for calcination bentonite was fired in furnace to high temp ( 700oC to 800oC) until Montmorillonite structure destroyed. Sulphuric acid was used in Acidification to treat calcined bentonite. XRD and XRF analysis carried out for the prepared samples by the three methods, sieving, calcination and acidification. Montmorillonite peaks were observed in raw and beneficiated samples,



with higher peaks after beneficiation. Calcination causes the peaks of sodium Montmorillonite to disappear leaving only one peak for magnesium Montmorillonite. Others Ions (Na, Mg and k) destroyed rather than ca-montmorillonite, which indicated bentonite to be calcium type. Sulphuric acid treatment removed the ferric oxide content [19].

Muazzez Karakaya et al.(2011) studied and characterized sodium and calcium bentonite located in Eastern Black Sea, Turkey. Montmorillonite was the main mineral, with slight existence of quartz, feldspar and biotite. Properties and characteristics of Na-bentonite and Ca-bentonite were studied in term of chemical and physical properties such as viscosity, swelling, gelling strength and pH. Clay particles less than 2 micron was extracted by sedimentation of samples in deionized water.

XRD showed that Montmorillonite is the major mineral in the samples, and very little kaolinite. Some samples showed high Na<sub>2</sub>O/CaO ratio (1 to 3) which is an indication to have high swelling capabilities. Many tests were conducted to test properties of bentonite. Viscosity was measured based on API RP 13D by mixing 22.5 gm of bentonite in 350 ml distilled water, also filtration at 100 psi [24].

JAMES et al. (2008) evaluated Yola Clay deposit in North-Eastern Nigeria, for drilling fluid applications. Dry and wet method were used for beneficiation, method and sodium treatment [23].

Bentonite in Nano-Particles size was evaluated in drilling fluid by Abdou et al.(2013). They studied local Egyptian bentonite and nano-bentonite grinded from those raw samples to a size of 4 to 9 nm. Results were compared to the standard bentonite used in

drilling fluids. Analysis of X-ray diffraction (XRD) and X-ray fluorescence (XRF) confirmed that the raw bentonite is mainly montmorillonite with small amount of kaolinite and quartz.  $\text{Al}_2\text{O}_3/\text{SiO}_2$  and Na/Ca ratio was 1/3 and 1.08%. Raw bentonite sieved with 63  $\mu\text{m}$  after treatment gave accepted performance in higher concentrations. Results comparison showed that nano-scale size of bentonite did not meet API specification <sup>[25]</sup>.

Karagüzel et al. (2010), upgraded raw bentonite from Turkey by treating it with soda and magnesium oxide, local Turkey bentonite collected and crushed into fine powder < 150  $\mu\text{m}$ , XRD analysis showed that it contained 50-55% Montmorillonite, with other impurities, feldspar 10-15 %, amorphous material 10% and some quartz and calcite 5-10% each. Non-treated bentonite showed bad performance and did not fulfill API standard. After activation with 1.5-3 % soda and 0.5 MgO the properties of bentonite enhanced significantly and can be used as drilling fluid according to the API standards <sup>[26]</sup>.

Very few studies were conducted on local Saudi bentonite, from different places, M.H. Al-Qunaibit et al.(2005)<sup>[27]</sup>, collected bentonite samples from the Jeddah area, located in the south-western region of Saudi Arabia. They studied mineral and chemical composition to investigate kinetics of sorption of  $\text{Cu}^{2+}$ . Study conducted by Al-Homadh(2007)<sup>[4]</sup>, showed that properties of local Khulays bentonite can be improved significantly by adding small amounts cheap materials used to enhance bentonite. Adding 5 % of sodium carbonate (soda ash) and adding cheap polymer such as Drispac at a very low concentration of 0.5 % increased viscosity by 200 % and decreased filtration by 25%. The prepared mud was 7wt% bentonite. Compared with 7wt% of commercial bentonite,

the viscosity and the filtration loss was nearly identical with the 8 wt% of enhanced local bentonite mud

#### **2.2.4 Bentonite Activation and Soda Ash Treatment**

Raw bentonite in natural form without treatment may not be qualified as drilling fluid additives even when it is rich in montmorillonite or good sodium quality, and may not deliver the required functions such as viscosity and filtration control <sup>[20]</sup>.

Bentonite performance and properties can be enhanced by applying different methods of treatments, such as inorganic additives. Addition of soda ash is well-known process to increase bentonite quality <sup>[26]</sup>.

Only 2 to 4wt% weight can significantly enhance bentonite properties, and it has been used in industry for decades. Sodium content greatly affects characteristics of bentonite. Na/Ca ratio affects physical properties of bentonite, (Foster 1953)<sup>[5]</sup>.

### **2.3 Challenges in Drilling Fluids (shale, deep drilling, and optimized rheology)**

Drilling in shale formation presents great challenge due to many problems associated with the swelling shale characteristics which result in high potential for wellbore instability <sup>[28]</sup>. Flat time in drilling operation caused by unstable wellbore problems such as tight spots in hole, hole enlargement, excessive solids (well packing), and bit balling. These problems are estimated to contribute by 20 to 30 % of drilling cost, and shale related instability forms 80 to 90 % of the root causes of this wellbore instability <sup>[29]</sup>, and shales form over 75 % of worldwide drilled formations <sup>[30]</sup>.

Before drilling, subsurface rocks are in chemical and physical equilibrium. The nature condition of shale when it is formed by sedimentation has particular arrays of clays, containing water with certain salinity and cation charges, and certain in situ stress state. After drilling this equilibrium will be disturbed, mechanically by cutting amount of rocks and by introducing drilling fluids into the previously balanced system <sup>[31]</sup>.

Wellbore stability is disturbed when imbalance between the rock stress and strength occurred due to mechanical and/or chemical mechanisms. Shale formation interaction with drilling fluid affect these mechanisms by different ways, capillary forces, osmosis phenomena, hydraulic, swelling and pressure diffusion are the general controllable factors. Imbalance in strength and compress forces happens when stresses are altered at near-wellbore shale formation, the interaction with drilling fluid alter its strength when pore pressure increased as filtration enters into the formation <sup>[30]</sup>.

Shales are sedimentary rocks with fine grains and extremely low permeability, and contains large amount of clay minerals. Water causes shale hydration and swelling which lead to deformation of rocks <sup>[29]</sup>.

Shale instability is attributed to content of reactive clays such as smectite, formation can react with drilling fluids and fall into the well causes cleaning problems, swell and decrease wellbore radius. Bedding and thin laminated shale can crack the rocks in various directions.

Shale reacts differently with drilling fluids due to different shale features such as clay minerals, rock structures, and deformation properties. Some of the methods used in the

laboratories to evaluate the impact of shale in wellbore instability are dispersion test, bulk hardness, and swelling test <sup>[28, 30,31]</sup>.

Based on laboratory studies conducted by S. Gomez et al. (2012), additives in drilling fluids with different chemical composition and concentrations have serious impact on fluid shale interaction and significantly control and reduce it.

Gomez et al.(2012), considered the available laboratory methods used to understand the deformation characterizes of shale formation, and found that immersion test can observe the physical and chemical changes of shale to give comprehensive idea of rock properties such as composition, structure and rock deformation features <sup>[28]</sup>.

Types of clays that react with water based drilling fluids and adsorb its water are kaolinite, illite, chlorite, montmorillonite and smectite. Other non-clay contents and types of clay that doesn't react with water such as quartz, feldspar, dolomite, calcite, siderite and gypsum may also cause mechanical instability problems, shale encountered in drilling are classified into 3 types <sup>[32]</sup>:

- 1- Brittle or sloughing shale, this type of shale has low montmorillonite content, but high kaolinite and illite fractions.
- 2- Gumbo or plastic shale, this type of shale consist of 10-25% montmorillonite, 20-30% of illite and possibly some formation water.
- 3- Hydratable or swelling shale, the type of shale have high content of montmorillonite and almost no formation water existence, this types because of montmorillonite contents can leads to excessive drilling fluid viscosity.

To reduce shale problems, the same level of salinity should be maintained in formation water and drilling fluid. The same concentration of the total dissolved solids should be maintained, and the same ion types in shale should be used in drilling fluids <sup>[32]</sup>.

Water based drilling fluids when used to drill shale formation tends to replace native pore fluids with an inhibitive fluids which causes a reduction in hydration stress and lower the ensile stress in surrounding walls of the well. The ratio between the rate of pressure destabilization and inhibitor fluid transportation into the shale is the key factor in controlling affectivity of inhibitor fluids <sup>[33]</sup>.

Shale stability should be investigated when using water based mud to evaluate water transport parameters such as flows driven by hydrostatic pressure, and chemical failure potentials. Interaction of drilling fluids with shale should be minimized as possible <sup>[28]</sup>.

Shale can form an effective membrane for osmotic flow of water; the same level of salinity should be maintained in formation water and drilling fluid. Instability can be controlled by reducing hydraulic inflow of mud filtrate by chemical additives that reduces effective shale permeability and plug pore throats with chemical agents or by using high mud filtrate viscosities <sup>[34]</sup>. The same dissolved solids, and same ion types in shale should be used in drilling fluids (Talabani et al 1993) <sup>[32]</sup>,

The problems of water-based drilling fluids with shale can be generally described in two main factors; the reactivity of clay to water and the stress of the rock caused by pressure propagating from the mud hydrostatic pressure. Based on study results for experimental data carried out by W. Rogers (1948), on the interactions of shale and water in drilling due to activity of water flow in or out of shale, this amount of water is affected by

different drilling fluids and additives, differential pressure or overbalance drilling and the moisture content which affect shale strength<sup>[35]</sup>.

Inhibitive drilling fluids are used to chemically stabilize the surrounding rocks and control chemical reactions of drilling fluids with shale formation, weakening rocks and decrease stress strength which lead to mechanical failure<sup>[29-31]</sup>.

Dispersion of solids in drilling fluids in addition to rock structure study is important to evaluate mechanisms of shale instability, experiments conducted by Sérgio et al. (2002) described an integrated methodology to characterize shales, in term of mineralogy, solid particles, pore fluids and microstructure of the shale, to identify and evaluate reactivity of the shale. Based on this study, the instability depends on pore geometry and shale structure for drilling cutting in addition to ion transport through shale<sup>[36]</sup>.

Water invasion into the shale formation during drilling should be controlled to control wellbore stability. Previous studies confirmed that water invasion to shale formation can alter the stress distribution of the formation by changing reducing rock strength and change young's modulus which cause mechanical failure of the wellbore<sup>[37]</sup>.

Common practice to enhance wellbore stability is to increase the drilling fluid salinity, but previous studies showed that high salinity in many cases lower the water activity. In drilling condition where faults or fractures are encountered the higher salinity is not preferable for stable wellbore, hence, optimized salinity is required, quantitative measurements of water ion movement can optimize the salinity of drilling fluid. (. Rojas et al. 2006)<sup>[38]</sup>.

Zang et al.(2006)<sup>[38]</sup>showed that osmotic property due to different in salinity is a valuable tool to control wellbore stability caused by water activity, but if overused it may lead to more instability problems. They also modified a method to calculate in-situ water activity of shale formation based on concept the effective mean of stress.

Using anions such as alts of alkaline or earth-alkaline metals and/or sulfonic acids were introduced to drilling fluids as shale stabilizer and loss control through a patent by Stefano Carminati(2000)<sup>[36]</sup>. Shale behavior under effect of different anions in water-based drilling fluids were studied by Carminati et al. (1999), properties such as viscosity, pore blocking mechanisms and osmotic phenomena was evaluated under high pressure and high temperature (HPHT) condition. Found that glycol effect in viscosity and pore blocking is negligible, but increases cutting hardness when used with potassium salts. Potassium based drilling fluids has more effect to the osmosis property <sup>[36]</sup>.

Oil based mud has a balanced chemical potential with shale formation, and used instead of water based mud to avoid instability problems (Methven et al. 1971)<sup>[31]</sup>, because it doesn't filtrate into shale if a certain threshold pressure is not exceeded <sup>[33]</sup>.

Rheology of drilling fluids is an important parameter and should be maintained under various drilling conditions in addition to filtrate loss. Vikas et al. (2004) developed a drilling fluid with better rheological properties. Analysis of drilling fluid filtrate impact on formation damage showed minimum damage in sandstone cores. Bentonite used in formulating the drilling fluids was obtained from the Kutch region, Gujarat, India. Raw samples were dried in sun for a few days, crushed and screened through 200-mesh size sieve (0.074 mm), and high viscosity polyanionic cellulose was obtained. Bentonite water



suspensions were then prepared at different compositions to measure the rheological properties such as apparent viscosity, plastic viscosity, initial gel strength and 10 min gel strength <sup>[39]</sup>.

Based on these results the ideal bentonite concentration was selected for further treatment, environmentally-friendly organic polymers was added to the bentonite suspension and rheological properties was measured again, thermal stability obtained after hot rolling in roller oven for 16 hrs. Formation damage was investigated based on permeability reduction in a sand stone core sample. They found that very low concentrations of tamarind gum with PAC produce favorable rheological properties and optimum fluid loss <sup>[39]</sup>.

Drilling fluids formulation for deep-water drilling present unique challenges, conditions in deep wells are very harsh in term of pressure and temperature. The pressure increases depend on fluid density and water depth, and temperature varies 1°C up to very high degrees.

The assumption of rheology profile to be independent of pressure and temperature is valid only in shallow wells where temperature change is not very large, so prior to drill a HPHT section, the rheological properties of proposed mud should be evaluated at HPHT conditions.

Higher temperature raises the concern about stability of drilling fluids under these extreme conditions. Rheological properties under this compression and expansion need to be maintained. A flat-rheology drilling fluid is designed to overcome the HPHT conditions of drilling. Rheology modifiers and viscofiers can hold within certain

operation conditions, so for HPHT drilling fluids formulas must be formulated to assure good temperature dependent rheology profile.<sup>[50]</sup>

Rommetveit et al. (1997) conducted experiments on drilling fluids at typical HPHT wells to determine the effect of pressure and temperature on mud rheology. In the experiments, different share stress and shear rates were applied, temperatures varied from 50 up to 200°C, found that at low shear rate it's less pressure and temperature dependent compared to the high shear rate. The dependence was higher and much more pronounced in oil based mud OBM than water based mud WBM <sup>[40]</sup>.

Davison et al. (1999)evaluated different drilling fluids systems, such as oil base mud (OBM), synthetic base mud (SBM) and water base mud (WBM). Oil based mud was highly affected in low temperature, and all the fluids thicken considerably at the very low temperatures.

OBM and SBM showed higher viscosity by 200% at low temperature the ration of oil/synthetic to water is changed by 10%.

water based mud showed a relatively larger increase in viscosity for the un-weighted mud salt/polymer-based fluids, the effects of increasing pressure at low temperatures did not significantly affect the salt/polymer-based fluids except for the weighted sodium silicate and potassium formate fluids which exhibit an increase in the fluid shear stress <sup>[41]</sup>.

Effect of rheology in rate of penetration was addressed by Beck et al. (1995); they collected field data and analyze the data to identify the impact of drilling fluid rheology in penetration rate. For good assessment all main parameters that affecting penetration rate selected as the optimized design such as hole size, weight-on-bit, rotary speed, bit

type, formation type, bit hydraulic energy, and basic mud type , to eliminate its affect. Eventually direct relationships were found between penetration rate and plastic viscosity. Penetration rate was increased by 58% when fluid rheology was adjusted <sup>[42]</sup>.

## **CHAPTER 3**

### **OBJECTIVES AND STATEMENT OF THE PROBLEM**

#### **3.1 Statement of the Problem**

Bentonite is one of the main materials used in drilling fluid formulation. Oil companies are consuming enormous amounts of bentonite in drilling and cementing applications in oil and gas wells. Saudi Arabia is the major oil producer in the world and its huge drilling operations demand for enormous amount of bentonite, over 100,000 tons a year, all of this bentonite is imported from abroad suppliers.

#### **3.2 Objectives**

The objective of this research is to study local Saudi bentonite and evaluate its application as drilling fluid additives and the specific objectives are as follow:

- 1- Define the compositional variation of the local Saudi bentonite from standard drilling grade one.
- 2- Study the properties of local Saudi bentonite and compare it with the drilling grade bentonite.
- 3- Enhance its properties using soda ash treatment
- 4- Evaluate its performance in drilling formulation at both low pressure/low temperature and high pressure/high temperature HPHT

### 3.3 Approach

The experimental study will be firstly conducted for non-treated raw bentonite to define the gap between local and drilling grade bentonite properties, which specified by API and OCMA, then small percentage of soda ash since it is a cheap additive will be considered to upgrade raw local bentonite properties to enhance its performance in drilling fluids.

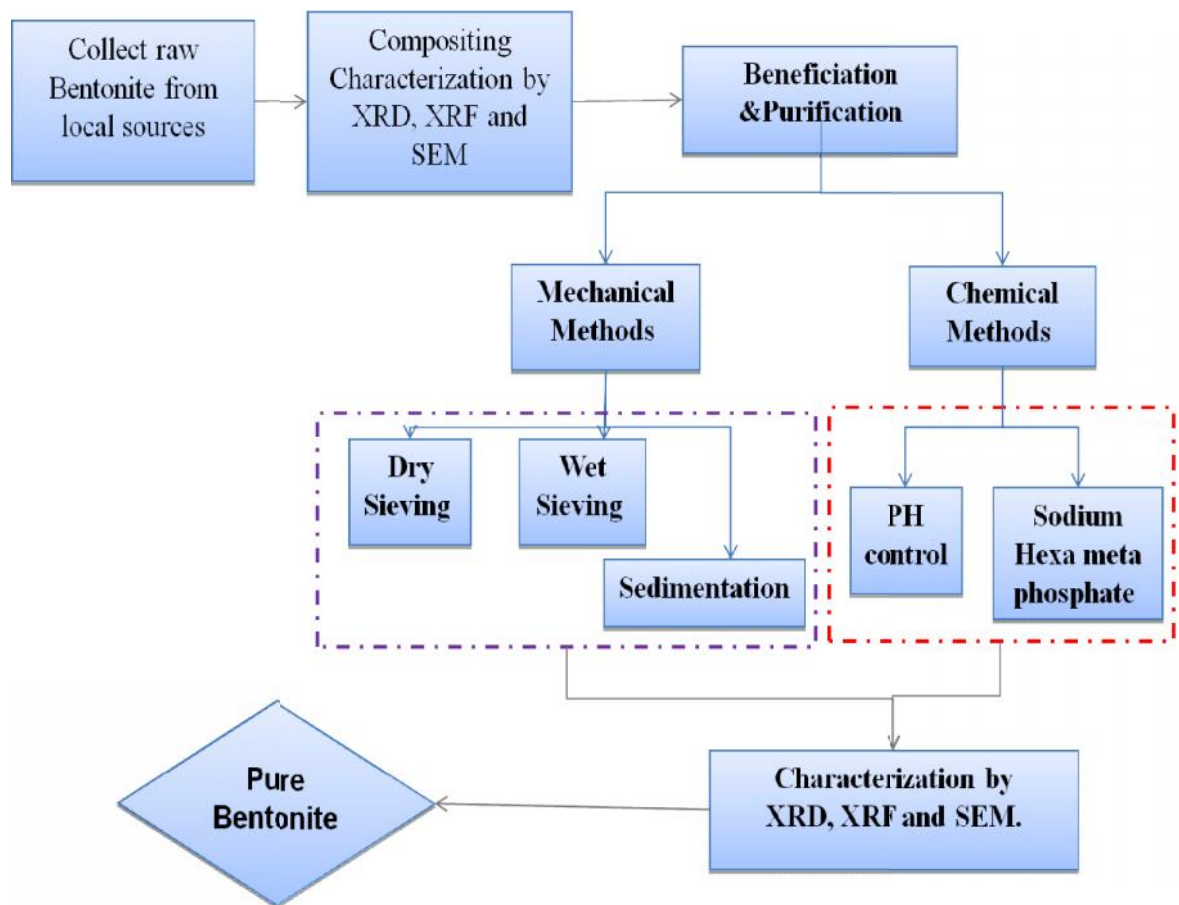


Figure 2 Beneficiation and Purification Methods of Raw Bentonite

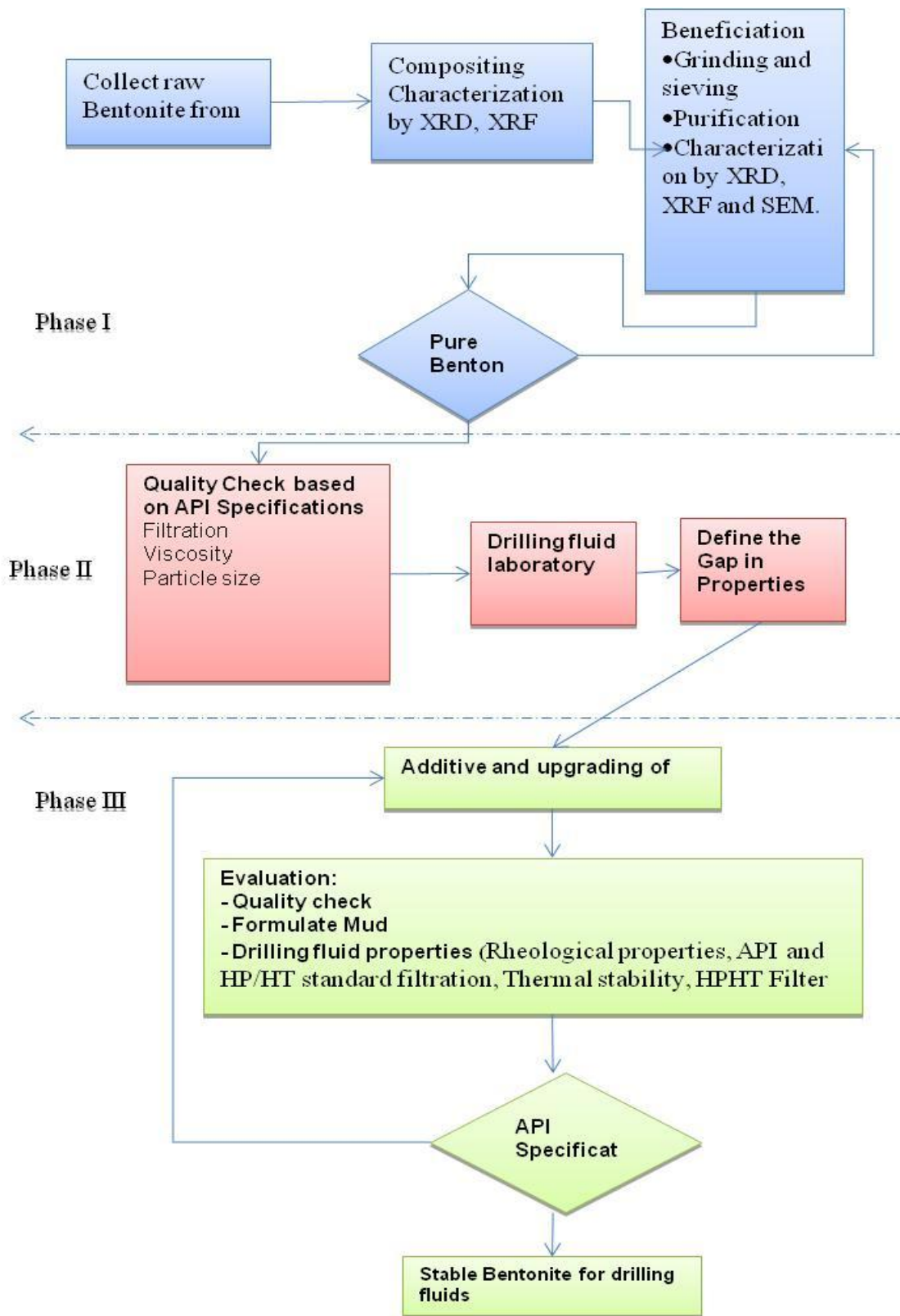


Figure 3 Experiments Flow Chart

# CHAPTER 4

## METHODOLOGY AND EXPERIMENTAL

### PROCEDURES

#### 4.1 Quality Check for Bentonite

Drilling-grade bentonite should meet the requirements of international standards. Some tests called quality-check-tests are performed. Montmorillonite clay in drilling fluids forms a gel like structure at low solid content 4-5 %w.(Rosi et al. 199)<sup>[43]</sup>. Percentage of Montmorillonite, > 80 % is preferred.

**Table.3** shows the physical specifications (API 13I recommended practice)<sup>[44]</sup>

**Table 3 API 13I Bentonite Physical Specifications**

Requirement	Standard
Viscometer dial reading at 600 r/min	30 Minimum
Yield point viscosity ratio	3 Maximum
Filtrate volume	15 ml Maximum
Residue of diameter greater than 75 micron	mass fraction 4 % Maximum

#### 4.1.1 Rheology Quality Check of Bentonite

Bentonite suspension in distil water should satisfy API requirement described in **Table.3**. The viscometer dial reading at 600 r/min should be above or equal 30, and yield point not more than 3 (API 13I recommended practice)<sup>[44]</sup> and Oil company materials association (OCMA) specification. Procedures for the test described by the following <sup>[44]</sup>:

Equipment

#### Rheology Test Procedures (API RP 131 lab testing)<sup>[44]</sup>

Sample is prepared at room temperature by adding 22.5 gm of bentonite to 350 ml of deionized water while stirring in mud mixer, any bentonite at the wall of the container is scraped using spatula after each 5 min to make sure all of it is suspended in the mixture, and stirring should be continued for 20 min.

Bentonite mixture was aged for 16 hrs. at room temperature prior testing by the viscometer. Before that sample should be stirred on the mixer for 5 min then put into viscometer cup at room temperature, the dial readings at 600 rpm and 300 rpm should be recorded when reading is stabilized at each rotational speed.

Calculations

Plastic viscosity = (Reading of 600 rpm – Reading of 300 rpm)      cP

Yield point = Reading of 300 rpm – Plastic viscosity      lb/100ft<sup>2</sup>

Yield viscosity ratio =  $\frac{\text{Plastic viscosity}}{\text{Yield point}}$



#### **4.1.2 Filtration Quality Check of Bentonite**

Low pressure/low temperature API filtration test is also used to evaluate bentonite quality. The suspension of bentonite in deionized water should not give more than 15 ml filtration volume as spurt loss. Sample is mixed with the same procedure by adding 22.5 gm of bentonite to 350 ml deionized water, mixed for 20 min and aged for 16 hrs. The spurt loss is measured by the following procedures:

##### **Filtration quality check procedures (API RP 131 lab testing)<sup>[44]</sup>:**

Sample of Suspension bentonite after aging is stirred for 5 min, and then poured into API filtration cell, a pressure of 100 psi is applied for 30 min while collecting the filtration from the bottom drain in a graduated cylinder, good bentonite should not filtrate more than 15 ml.

#### **4.1.3 Particle Size Quality Check**

Bentonite particle sizes are in the micrometer to nanometer scale, montmorillonite particles are less than 2  $\mu\text{m}$ . API and OCMA specifications recommended that drilling grade bentonite should have particle size less than 75  $\mu\text{m}$ . The residue that retained by 200 mesh should be less than 4% of when 50 gm of bentonite is sieved. The detailed procedure as follow:

##### **Sieving analysis procedures<sup>[44]</sup>:**

A sample of bentonite is weighted 50 gm using a balance. Two sieves, 100 mesh (150  $\mu\text{m}$ ) and 200 mesh (75  $\mu\text{m}$ ) are cleaned properly, put into moisture oven at 85°C to remove the humidity for 30 min. Bentonite samples is put in the 100 mesh

tray at sieve shaker machine and time is set for 10 min, at least 96% wt should pass the 200 mesh (75  $\mu\text{m}$ ) and be collected at the bottom tray. Only maximum of 4 % wt is retained and collected at 100 and 200 mesh sizes.

#### **4.1.4 Particle Size Distribution (PSD)**

Drilling grade bentonite is recommended to have uniform particles size distribution to avoid settlement of the larger size and phase separation. Generally in drilling fluid particle size is important to determine whether there will be erosion or formation damage if trapped in the formation pores and whether it will control fluid loss and allow effective bridging in mud cake. Particle size analysis can be carried out using sieve shaker shown in **Fig.4**.

In the vibrating sieve shaker, sample is thrown upwards by the vibrations of the sieve bottom and falls down due to gravitation forces. By vibrating the sample is subjected to a 3-dimensional movement. Particles are accelerated in vertical direction, rotate freely and then fall back. The sample material is spread uniformly across the whole sieve area. PSD also can be analyzed using a laser scattering particle size analyzer, shown in **Fig.5**.



**Figure 4 Vibrating sieve shaker**



**Figure 5 Particles size analyzer Laser diffraction**

#### **4.1.5 Clay Mineralogy Characterization**

Type of clay is defined by its minerals and Ions, bentonite has high montmorillonite content, and other clay types are illites, kaolinites and chlorites. Pure and high quality bentonite has the higher percentage of montmorillonite. X-ray diffraction is used to determine the mineralogical composition. Sample exposed to X-ray radiation, the diffraction pattern is compared to the known standards patterns of elements to determine contained minerals in the sample.

SEM (scanning electron microscope) shows the microstructural changes in clays with high spatial resolution

#### **4.1.6 Viscosity and Gel Strength**

Viscosity and Yield point and gel strength can be measured using Fan viscometer (Direct-indicating viscometer FAN-35). It is a rotational instrument powered by an electric motor, it allows speeds of 3, 6, 100, 200, 300 and 600 rpm, the reading from rotor sleeve speeds of 300 and 600 rpm is used to determine plastic viscosity and yield point, and 3 rpm is used to determine the gel strength.

#### **Procedures (API RP 13 B-1 4<sup>th</sup>ed 2009)<sup>[44]</sup>:**

- Shake the mud sample and properly pour it into the cup until it reaches the indicated line.
- Place the container and immerse the rotor sleeve exactly to the scribed line, the sample will be in the annular space between the two concentric cylinders
- Record the temperature of the sample
- Run it at 600 rpm and wait for the viscometer dial reading to stabilize at steady value
- Reduce the rotor speed to 300 rpm and when stabilized record the dial reading
- Increase the speed to 600 rpm and allow the sample to stir for 10 sec then stop the rotor and wait for 10 s.
- Run at 3 rpm and record the maximum reading in pounds per 100ft<sup>2</sup> (1 Pa =0.511 lb/100 ft<sup>2</sup>)

- Again stir the sample at 600 rpm for 10 s, then stop the rotor and wait for 10 min before re-stir at 3 rpm and record the maximum dial reading.

### Calculation

Plastic viscosity = Reading of 600 rpm – Reading of 300 rpm

Yield point = Reading of 300 rpm – Plastic viscosity

$$\text{Apparent viscosity} = \frac{\text{Reading at 600 rpm}}{2}$$



Figure 6 Viscometer

### 4.1.7 API Filtration

Filtration test measures the amount of mud invasion into the permeable formation and the filter cake that will form on the wall of the wellbore when filtration takes place. API filtration test is conducted at room temperature

## **Equipment**

Low-temperature/low-pressure API filter press, cell inside diameter 76,2 mm (3 in) height at least 64,0 mm (2,5 in), filtration area 45.8 cm<sup>2</sup> (7.1 in<sup>2</sup>)

## **Procedures (API RP 13 B-1 4<sup>th</sup> ed 2009)<sup>[44]</sup>**

- Clean the cell properly and dry it, place a new clean and dry filter paper and make sure the gasket are in good condition
- Close the relief vale and adjust the pressure to 100 psi (690 kPa), reach the pressure within less than 30 s, test period begins when pressure is applied.
- Collect the filtrate in a dry graduate cylinder from the bottom of the tube drain
- Keep applying the 100 psi pressure for 30 min
- Measure the volume of filtrate in milliliters. Shut off the flow through the pressure regulator and open the relief valve carefully.
- Make sure all pressure has been relieved, then remove the cell from the frame
- Take the filter paper containing the mud cake, wash it gently with water, and measure its thickness to the nearest millimeters (1/32 in)



**Figure 7 Low-temperature/low-pressure API filter press**

## **4.2 Water Based Drilling Fluids Laboratory Tests**

### **4.2.1 Drilling Fluid Density (mud weight)**

Mud weight is measured using the mud balance, either pressurized mud balance if there is a foam or gas in the mud, or the conventional one. The balance is equipment designed to hold a cup full of mud and balance it using a sliding weight along graduated beam. A level-bubble is used to horizontally align the balance for accurate reading. Mud balance should be calibrated frequently with fresh water to give a reading of 1,00 g/m or 62.35 lb/ft<sup>3</sup>.

**The detailed procedure are as follow:(API RP 13B-1 4<sup>th</sup>ed 2009)<sup>[44]</sup>**

- Set the instrument on a flat, level surface.
- Measure and record the temperature of the drilling fluid.
- Make sure the cup is cleaned and dry, fill it with the sample and put the cap on top of it, hold and rotate the cap on top of the cup until it is firmly seated.

Ensure that some of the drilling fluid is expelled through the hole in the cap in order to free any trapped air or gas.

- Hold the instrument from the beam and wipe the outside of the cup until it is clean and dry, while covering the cap hole to avoid disturbing of the sample.
- Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the centre line.
- Read the drilling fluid density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of g/ml, lb/gal, and lb/ft or as a drilling fluid gradient in psi/1 000 ft.



**Figure 8 Pressurized Mud Balance**



**Figure 9 Mud Balance**



#### 4.2.2 PH

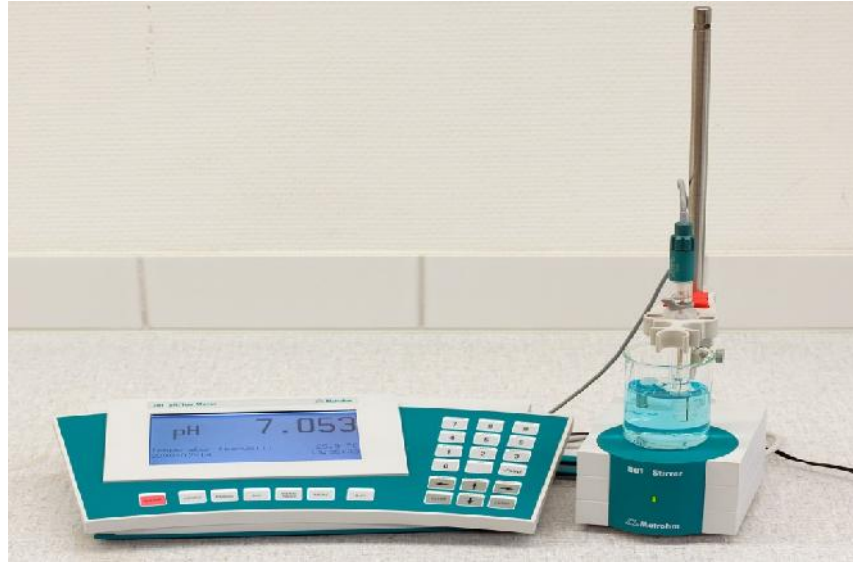
Drilling fluid pH is very important since it affects fluid loss and solubility of many additives; some of additives are effective within certain ranges. pH is measured for the drilling fluids and for the filtrate. The best way to measure pH is by using a glass electrode pH meters. pH papers and sticks are not recommended due to errors resulting from the nature of drilling fluids that containing solid, chemical and dissolved salts. The detailed procedures are described below.

##### Equipment

Glass-electrode pH meter measuring the potential between a glass-membrane electrode and a standard reference electrode, pH range from 1 to 14 accuracy  $\pm 0.1$ , and operating temperature range: 0 °C to 66 °C (32 °F to 150 °F). Instruments should be calibrated using set of three buffers of 4.0, 7.0, and 10 pH.

##### Procedures (API RP 13B-1 4<sup>th</sup>ed 2009)<sup>[44]</sup>:

- Make sure the calibration buffers and sample are at the same temperature (preferred room temperature).
- Rinse electrodes by distilled water and clean it dry
- Calibrate using 2 or three buffers (4.0, 7.0 and 10) until slope satisfied, rinse in distilled water and clean before each.
- After calibration rinse electrode again with distilled water, place it in the sample and stir gently.
- Wait 1 min for the reading to stabilize, record pH to the nearest 0.1 unit.



**Figure 10 PH meter**

### **4.2.3 Rheology**

Downhole conditions have wide range of pressure and temperature and drilling fluid exposed to this condition will be affected. Stability and viscosity profile can change accordingly. The rheological parameters such as plastic viscosity, apparent viscosity and yield point of the mud all changed with pressure and temperature, and these parameters control frictional pressure drop and solids-bearing capacity <sup>[45]</sup>

Rheological parameters are important in the calculation of circulating hydraulics, hole cleaning efficiency; orifice viscometer Marsh funnel viscometer can give an indication of viscosity, by measuring the time rate of flow. The orifice viscometers consist of a conical funnel that holds 1.5 liters of drilling fluid with an orifice at the bottom of the cone. Designed so that by the outflow time of one quart of fresh water at  $70\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$  ( $21\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ) is  $26\text{ s} \pm 0.5\text{ s}$ . (API RP 13D – Rheology and hydraulics of oil-well) <sup>[44]</sup>, procedures as follow:

- Clean and dry the funnel, then cover the funnel orifice with a finger and pour the sample through the screen until it reach the indicated level
- Put a clean graduated cup
- Remove finger and start stopwatch.
- Measure the time for drilling fluid to fill to 946 ml and report the time to the nearest second, as the Marsh funnel viscosity.

Marsh funnel is a quick indicator but not recommended in evaluating drilling fluids and comparison between different formulas and component. Using direct-indicating viscometer is recommended. Detailed procedures for Direct-indicating viscometer procedures described at previous section.

#### **4.2.4 High Pressure/High Temperature (HPHT) Rheology Test:**

High pressure and high temperature also alter the rheological properties of drilling fluid, rheology profile is determined by measuring all parameters using HPHT rheometer in different speeds and adopting one of non-Newtonian model, then plot shear stress versus shear rate. Test can be conducted using Fann Rheometer or Chandler HPHT model 5550.

Chandler HPHT model 5550 is associated with software that allow setup of desired range of speeds, ascending and descending profile, and measure rheological properties (Plastic viscosity and Yield) at each speed. Properties of drilling fluid is affected by to different share stress and will alter its properties especially viscoelasticity and thixotropy<sup>[46]</sup>.

The software of Rheometer fits the data points using Power law and Bingham plastic models. After sample is properly placed on instrument chamber, temperature and pressure is set to desired values and left to stabilize. A test schedule has to be

programmed for the values of speeds in rotational per minute, to allow the rheometer to read and record all parameters at each speed. The upward and downward speed schedule is to assess the thixotropic behavior of drilling fluid <sup>[47]</sup>.



**Figure 11 HP/HT Rheometer**

#### **4.2.5 Drilling Fluid Aging Test**

Time and temperature can cause degradation in component of drilling fluid and alter its properties and performance. Aging test is conducted to assess the effect of temperature in fluid properties. The fluid is left to age at elevated temperature for 16 hrs. Aging test can be conducted under static or dynamic condition; static aging simulates the case when fluid is left in the wellbore unmoved for long time during tripping, connection or other operations such as logging. The effect of dynamic aging is greater than static aging <sup>[48]</sup>.

Dynamic aging simulate drilling operation when fluid is circulated in and out under elevated temperature. The fluid is kept in a pressurized cell at the desired test temperature

with rolling panes allow the cell to roll or rotate while temperature is kept desired value for 16 hrs.

**Aging procedures[44]:**

- Pour the sample in aging cell, only 350 ml, equivalent to 1 bbl. should be filled, to allow for expansion  $\pm 2$  inches.
- Pressurize the cell to avoid evaporation
- Maintain desired temperature and allow rolling for 16 hrs.
- Take the cell out of oven and cool it to room temperature then release the pressure and open the cell.
- Conduct all drilling fluid evaluation tests such filtration and rheological properties, determine values of effective viscosity, plastic viscosity, yield point and gel strength (10 sec and 10 min)



**Figure 12 Rolling Oven and Aging Cell**

#### **4.2.6 HPHT Filtration**

High pressure High temperature test is conducted to assess drilling fluid filtration properties at condition similar to actual drilling condition, where the filtration occur at elevated temperature and pressure, under effect of back pressure from the formation.

Test is conducted using HP/HT filter press, the equipment allows applying pressure to filtrate and back pressure to simulate pore pressure and also to prevent evaporation of filtrate. Pressure is applied from pressure source CO<sub>2</sub> or nitrogen, pressure range from 600 up to 1300 psi, (API RP 13 B-1 4<sup>th</sup> ed 2009) <sup>[44]</sup>, in filtration test the difference between applied pressure and back pressure is maintained 500 psi.

**Procedures of the test as follow, (API RP 13 B-1 4<sup>th</sup> ed 2009) <sup>[44]</sup>:**

- Preheat the cell at 6 °C (10 °F ) above the desired temperature
- Condition the sample by stirring it for 10 min with high speed mixer. Then pour it into the sample cell.
- Cover the cell and close the upper and lower valves and lock in place
- Keep cell valves closed and apply 100 psi, the pressure is applied by adjusting top and bottom regulator
- Pressure should be applied in two steps, step one, apply 100 psi and maintain it for 1 hr, then increase the pressure to 600 psi and open the bottom valve to start filtration.

- Collect filtrate in a graduate cylinder for 30 min. (in high extreme temperature wait 5 min before opening the drain valve to allow cooling of filtration and avoid evaporation).
- Correct the filtrate volume. Because HP/HT press has filtrate paper of  $22.6 \text{ in}^2$  ( $3.5 \text{ in}^2$ ), it must be corrected to a filter area of  $45.8 \text{ cm}^2$  ( $7.1 \text{ in}^2$ ) by multiplying the filtrate volume.



**Figure 13 HP/HT Filter Press**

#### **4.2.7 Zeta Potential**

Zeta potential is a measurement of the surface charges at the particles in suspension. The stability of the suspension is directly related to the interactions of between the particles. Zeta potential was measured using Brookhaven zeta potential analyzer, This machine calculates zeta potential by measuring the electrophoretic mobility using laser Doppler method (ZetaPlus) and Phase Analysis Light Scattering (ZetaPALS).

Samples were prepared by dispersing 0.125 gm of bentonite in 50 ml deionized water. Homogenous and well dispersed suspension was obtained through shaking for 24 hrs.

Centrifuge was used to separate the larger particles as a quick sedimentation process and the top solution was drained by syringe needle and transferred into another containers. Before measuring ZP the samples were tested for particles size and pH using ZP instrument accessories. Minimum of 3 to 5 trials were run for each sample and calculation was done by the machine using a model relates mobility of the laser to the zeta potential.

#### **4.2.8 Hydrometer**

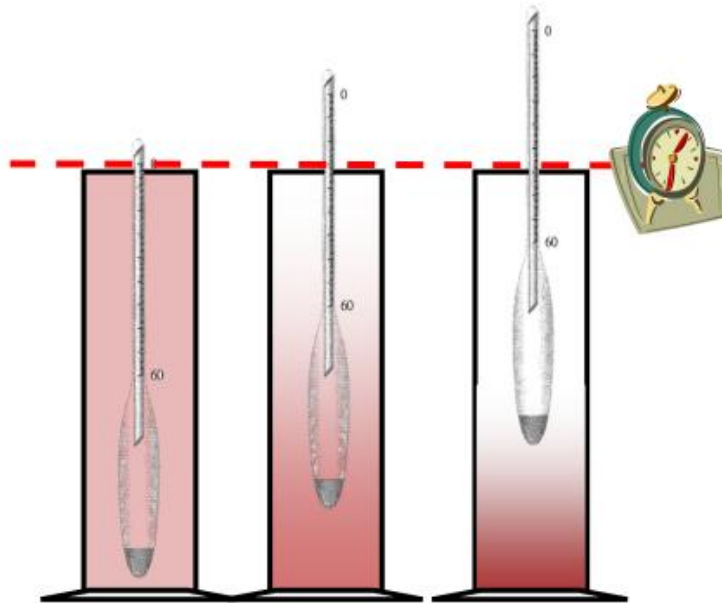
The percentage of clay, sand and silt in a rock sample was estimated through hydrometer analysis. The method is based on Stoke's law governing the rate of sedimentation of particles in a water suspension. The raw sample of clay is treated with sodium hexa meta phosphate to form aggregated solution full of cations that will keep the clay in a suspension state and all sand will precipitate.

##### **Procedures of the test as follow:**

- Mix a 50 gm of the raw clay that has passed through No. 200 sieve with 125 mL of 4% solution of Sodium hexa meta phosphate (deflocculating agent) in a 250mL beaker.
- Transfer the solution in the glass sedimentation cylinder and add distilled water to it up to 1000 mL mark, the solution can be mixed well by flipping the cylinder up and down with the top covered by a plastic bag.
- Obtain the hydrometer readings using the top of the meniscus on the hydrometer, for 2 readings within the first 2 minutes and then each 10 minutes for the second 2 hours, then for any suitable time intervals until the reading stabilized. Use a thermometer to record the temperature of the solution.



- After taking the reading, the hydrometer is placed back into the control solution with the same hexameta phosphate concentration and no soil in it.



**Figure 14 Hydrometer reading taken at different time**

## **CHAPTER 5**

### **RESULTS AND DISCUSSION**

This chapter discusses the results of Khulays bentonite processing and treatments, it describe in details the mining process, purification methods, and the chemical and physical characterization of the local bentonite. The results of soda ash activation are reported in details. It describes the significant improvement in swelling capacity resulted from the heating/stirring treatment. The impact of treatments on filtration, viscosity, particle size, swelling capacity and whole rheological properties of the local bentonite

#### **5.1 Local Bentonite Processing**

##### **5.1.1 Mining Process**

Local bentonite was extracted from Khulays deposit through mining process. Mining holes was selected at 8 different locations, to get a representative samples for Khulays bentonite. The topsoil was removed, it is only a few inches, consist of dirt and sand. The weathering effect appears at the surface of the overburden rocks. Weathering was in form of oxidizations and erosion. Some yellow layers and spots were observed in affected bentonite at the surface of overburden rocks. All affected rocks were scraped prior to get the fresh bentonite. Khulays bentonite color varies from light to dark grey and greenish brown color. The collected samples then examined and grouped into 2 sets, grey and greenish brown, Fig 15. There were some black particles in both of them which indicate existence of the oxide manganese. These two sets of samples were deliberated as

the target of the research, to identify the difference in physical and chemical composition. The samples were nominated as bentonite-1 and bentonite-2; both samples were fully characterized and taken through the purification process for comparison against the standard bentonite.



**Figure 15 Khulays raw bentonite**

### **5.1.2 Grinding**

Grinding of bentonite into fine powder was performed using ball grinder or milling machine. Firstly, large size rock ( $> 20$  mm) of Khulays bentonite was crushed using metallic hummer. Also a jaw crusher might be used for crushing the rocks into small pieces; so that it can be easily handled by the grinder. Any yellow spots of oxidization or erosion were eliminated. The chips were grinded using milling machine.

Grinding time greatly affects bentonite properties, and results in different particles sizes. In drilling fluids the required bentonite particle size has to have a uniform distribution, with range of  $2\text{ }\mu\text{m}$  to  $75\text{ }\mu\text{m}$  and the average should be around  $10\text{ }\mu\text{m}$ . when grinding

time was set to be too long (2 hrs for ball grinder) the resulting particle size was around 4  $\mu\text{m}$ . This size is not matching the standard bentonite particle size distribution. Grinding time of 20 minutes was sufficient. For the milling machine only 3-5 minutes gave the required degree of grinding. Dry sieving through 200 mesh was used to take the proper range of particles ( $<75\mu\text{m}$ ). The inputs of the milling machine has to be of small size (20 mm > particles > 2mm). The grinding was done for two types of samples. Before purification the raw bentonite rocks which crushed by hummer and after purification the samples taken from the evaporating dishes in form of crispy pieces.

### **5.1.3 Purification**

Raw bentonite which extracted through mining process is expected to contain many impurities and non-clay materials. Purification of bentonite is aiming at getting rid of all these contamination and obtaining a pure montmorillonite. From observation and analysis, Khulays bentonite contains some sand, quartz, feldspar, and iron. Impurities can be removed mechanically or chemically, it depends on the unique suspension property of the bentonite and its particle size. A set of purification methods were tried out and evaluated in term of effectiveness, recovery, cost and time. The most convenient way was the wet sieving after sedimentation for 24 hrs. Some of these methods are listed here:

- i- Dry sieving
- ii- Wet sieving
- iii- Sedimentation in deionized water
- iv- Sedimentation with 1 % sodium hexa meta phosphate
- v- Sedimentation/sieving combined method.

### 5.1.3.1 Dry Sieving Method

Dry sieving was done for Khulays bentonite through set of sieves with different sizes to know what kind of materials retains in each size. It also helps in generating mechanically particle size distribution. Before grinding samples were put into oven for 16 hrs. at 105°C to get rid of the humidity. For bentonite-1 sample, the weight before drying was 5064.5 gm and after drying become 4650 gm. That means the sample lost about 8% of its weight as water, this value is acceptable as the common water content in bentonite which is vary from 5% to 10%. Bentonite-2 also contained 8.5% water, after heating 4501.1 gm at 105°C for 16hrs. it lost about 382 gm of water as shown in **Table.4**.

**Table 4 Humidity removal before grinding**

Sample ID	Weight before drying gm	Weight After drying gm	Water loss gm	Notes
Bentonite-1	5064.5	4650	414.5	8% water lost at 105°C for 16 hrs
Bentonite-2	4501.1	4119	382.1	8.5% water lost at 105°C for 16 hrs

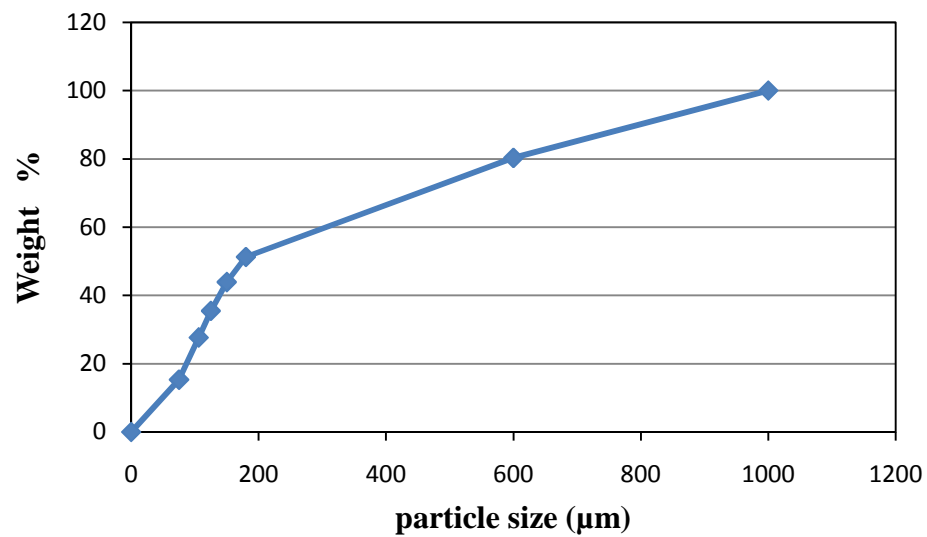
After drying, samples prepared for dry sieving by reasonable grinding using milling machine, firstly crushed using metal hammer. Grinded bentonite collected and a known weight of bentonite put into a sieve shaker top tray. Different sieve sizes were used to obtain the particle size distribution as 600 µm, 180 µm, 150 µm, 125 µm, 106 µm and 75 µm.

Due to shaking and movement applied by the shaker the sample material was spread uniformly at the trays and fractionated based on its different particles of sizes. Targeted bentonite particles are considered to be less than 75 micron. Only 15% of the grinded sample was recovered through mechanical dry sieving and nominated as A-1. The results are shown in **Table.5**.

Dry sieve recovery can be increased if bentonite is grinded into very fine grains, but the separation will not be effective. The impurities particles will be grinded to the same as bentonite particle size and will pass through the sieve. Fig.16 shows the particle size distribution of dry sieved samples, only 15wt% passed through 200 mesh sieve, which indicate that 85wt% of the bentonite will be falsely considered as impurities. Bentonite particles are sticky and hold to each other and to the larger particles, which prevent them from passing through the sieve. In the bottom tray, observed that it contains sand and silt, the color changed from grey to off-white. Further investigation by sedimentation verified that obtained powder is settling quickly after only 5 minutes when dispersed in water. This is attributed to that sand and silt particles are very small and heavier which make them easier to separate through mechanical fractionating. Shaking and movement applied by the machine separated these heavier particles. Dry sieving will leaving most of the bentonite particles on the upper trays, therefore other methods should be considered.

**Table 5 Dry sieved bentonite, particles size distribution**

<b>Particles Size Range</b>	<b>Bentonite-I Weight (gm)</b>	<b>weight %</b>
Particles > 600 $\mu\text{m}$	975.5	19.72
600 > size > 180	1435	29.01
180 > size > 150	363.9	7.36
150 > size > 125	417.8	8.45
125 > size > 106	385.5	7.79
106 > size > 75	611.3	12.36
passed 200 mesh (size < 75 micron)	757.6	15.32
Total sample weight	4946.6	100 %



**Figure 16 Particles Size distribution of the Dry sieved samples**

### 5.1.3.2 Wet Sieving Method

Wet sieving was used to overcome the stickiness of bentonite particles and increase the recovery percentage. Also to avoid grinding the impurities to a fine powder that can bypass through dry sieving. Rock samples without grinding were put into freshwater 1 to 5 by weight and left overnight until it dissolved completely. Then the suspension was poured on the upper tray of the sieves set. Gentle agitating was applied to help screening the particles through the sieves, and repeated at each sieve starting from the upper larger sieve to the lower smaller one. The sieves used were as follow; mesh sieve No.400 (38  $\mu\text{m}$ ), No.325 (63  $\mu\text{m}$ ), No.200 (75  $\mu\text{m}$ ). Samples collected through each sieve, weighted and nominated as shown in **Table.6** as S1, S2, S3, S4 and S5. The recovery of wet sieving was more than 85% of the sample, the obtained samples were put into an evaporation dishes for 24 hours to get a dry bentonite ready for the grinding. Sample of wet sieving less than 75  $\mu\text{m}$  were nominated as w-1. Particles size distribution is shown in **Fig.17**.

**Table 6** Wet sieving analysis

Sample ID	Particle size	Bentonite Weight (gm)	weight %
S1	size <38 $\mu\text{m}$	253.9	79.3
S2	63> size >38	22.6	7.1
S3	75> size >63	7.6	2.4
S4	150> size >75	12.9	4
S5	Very large particles >150 $\mu\text{m}$	23	7.2
	Total sample	320	100%



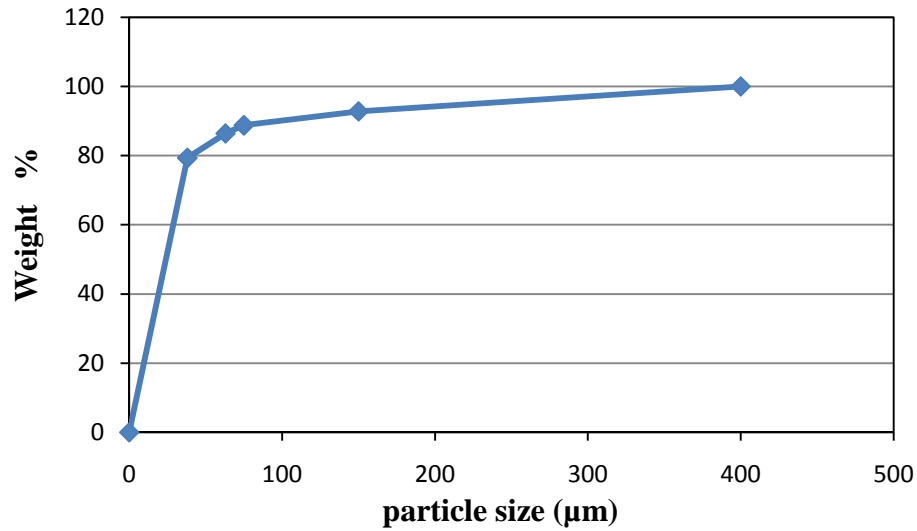


Figure 17 Particles size distribution of Wet sieved samples

### 5.1.3.3 Sedimentation Method

Purification through sedimentation methods depends on the suspension property of the bentonite. An inherited property of bentonite is to absorb water and expand, when bentonite is dispersed in water, it forms highly stable colloidal suspensions. Water viscosity will be increased due to swelling of the bentonite platelet when water molecules invade between them. When raw bentonite dispersed in water, any impurities of sand or quartz will not be suspended and will rapidly settle down. Suspension and sedimentation of the particles in water depends on many factors, such as particle size, pH, time, concentration and temperature. Understanding of the parameters affecting sedimentation process will define the suitable method for Khulays bentonite purification. Two different procedures were adopted in preparing the raw bentonite of Khulays deposit for sedimentation process.

- 1- Raw samples without grinding
- 2- Grinded and sieved samples (< 75 microns).

For the first approach, the raw bentonite was taken as rock pieces without grinding. A sample of 200 gm. was put into 2000 ml of distilled water in plastic container. The bentonite was dissolved in water using only hand with gentle wiping until all course dispersed into the water. This process will allow the bentonite sample to disperse gradually in water and leave the impurities such as sand, silt and quartz to settle down, and then stirred with magnetic stirrer for 3 hours. Solution left to settle undisturbed to allow sedimentation of large particles, but noticed quick phase separation and clear water layer in the top of the plastic container (shown if **Fig.18**).



**Figure 18 Phase separation after sedimentation for 5 minutes**

Quick separation maybe because of the wide range of the particle size in the solution or simply it's the calcium bentonite property which has bad swelling property. The calcium bentonite suspension is less and settles more rapidly than the sodium bentonite suspension.

The suspension was left undisturbed for 5 minutes, and then the supreme solution was collected. Bentonite suspension collected from supreme solution then centrifuged for 30 minutes to collect the particles and remove the water. The sample nominated as Sed-1.

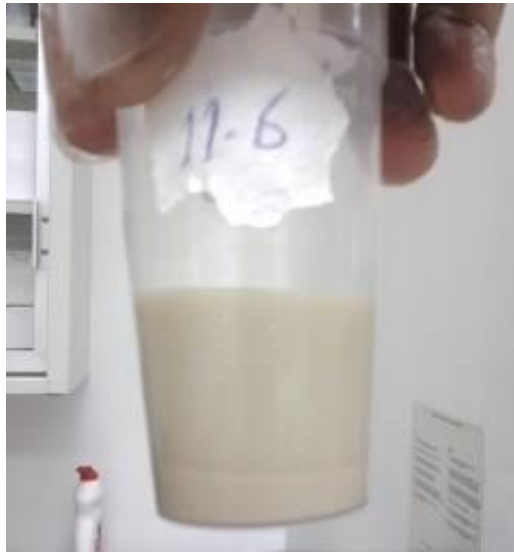
Recovery through sedimentation process is quite low (less than 5%), very little bentonite was extracted through centrifugal or drying of the supreme solution. Again this is because Khulays bentonite is a calcium bentonite and does not disperse in water as good as sodium bentonite. Also the wide particle size different does not allow for homogenous suspension. The larger particles are settled quickly due to gravity. Reasonable grinding is recommended before sedimentation, however care must be taken not to grind the rock to a level that impurities particles is near bentonite particle in size. Concentration of particles also important and should be set as low as possible.

For the second approach, raw bentonite was grinded to fine powder using a milling machine, and big particle removed. Grinded bentonite sieved through 200 meshes (75 $\mu$ m) to ensure all particles are within reasonable range suitable for sedimentation process. A sample of 200 gm. of bentonite < 75  $\mu$ m was put into 2000 ml distilled water. Solution concentration was 10%, solid to liquid ratio was 0.1 gm to 1 ml of distilled water.

The solution then left undisturbed and observed for 5 min, again a quick separation was occurred. This observation eliminates effect of the range of the particle size because the used sample is homogenous fine powder less than 75  $\mu$ m. Other parameters affecting the separation are pH of the solution and the concentration, the pH measured was 7.6, and concentration is 10%. For the same sample, solid to liquid ratio was decreased and solution set to concentration of 5 %. Also pH increased up to 11.6, then solution left undisturbed.

In higher pH, sedimentation took longer time, for pH range 7.6 (original), 10.1, 10.6, 11.2 and 11.6, separation slowed down from 5 minutes to 30 minutes leaving three distinct

layers. This indicates that in higher pH the calcium bentonite suspension is improved (**Fig.19**). The upper layer (took 50% of sample) is light water with little bentonite suspension, the same as the supreme solution occurred in non-grinded samples. The solution in the middle (40-45% of the sample) is bentonite with good suspension, at the bottom of the plastic container; some of the impurities were settled down. For all impurities to be removed the solution should be left undisturbed for 24 hours. A syringe was used to suck the bentonite solution out leaving the bottom layer undisturbed. Pure bentonite extracted and obtained using evaporation dishes.



**Figure 19 Raw bentonite suspension in high pH**

#### **5.1.3.4 Sedimentation With 1 % Sodium Hexa-meta Phosphate**

Hexa-meta phosphate method is based on invention made by Paul Schick in 1975. He used sodium polymeta phosphate ( $\text{NaPO}_3$ )<sub>n</sub> to extract pure bentonite with high quality. This method is used when sedimentation with distilled water is not an efficient and takes too much time. quartz, feldspar, ilite and sands are removed by 90% efficiency. To prepare 1:7 of bentonite to water solution a 200 gm of raw bentonite was dissolved in 400

ml of water, and then added to 1000 ml of hexa-meta phosphate solution. At the beginning 0.5% hexa-meta phosphate solution was prepared by mixing 10 gm of the poly phosphate in 1000 ml of distilled water and stirred for 5 minutes. The 400 ml of bentonite slurry was added slowly while the poly phosphate solution is stirred. The bentonite gradually dispersed into the solution and impurities separated and settled in the bottom of the container. The solution was left for 10 minutes which is enough of sedimentation process to take place. Observed solids and large particles layer at the bottom of the container, this is the impurities which is easy separated and above pure bentonite dried in evaporation dishes prior to be grinded. Recovery was high, more than 70% and time was efficient, only 10 minutes for all impurities to be separated. The pure bentonite obtained through sedimentation with 1% of sodium hexa-meta phosphate was nominated as hexa-1. This bentonite need to be washed many times with water to remove the sodium meta phosphate solution completely.

#### **5.1.3.5 Sedimentation/Sieving Combined Method**

This combined method is proposed for purification of Khulays bentonite to overcome some limitations in the methods mentioned in literature and being used in the industry. The drawback of dry sieving as a mechanical differentiation method is having a low recovery, wet sieving increases the recovery but increases the contamination as well. Sedimentation method is time consuming but gives good results if the others drawdown of the parameters that affect sedimentation are avoided, such as particle size wide range and low pH. Calcium bentonite bad suspension also presents another challenge. The method of sodium hexameta phosphate is overcoming the time consuming and suspension problem by helping bentonite to dispersed and remove any impurities within

only 10 minutes. But it's a complex process and requires additional cost for process make up and additional materials.

The sedimentation/sieving combined method is easy to be used in the laboratory and also can easily be designed for large quantity production in industry. It uses the efficient separation of the sedimentation method with the high recovery of wet sieving method. The raw bentonite is taken through a simple and non-costly process. Firstly it will be grinded to 75  $\mu\text{m}$ , the bentonite with the impurities will be in uniform particle size distribution to be easy for separation. The grinded bentonite is put into large plastic container to maintain low consecration ( $<10\%$ ). Only 2 to 6 hours is enough for all the impurities to be settled down. But before that the bentonite supreme solution which forms at the very beginning within the first 10 minutes will be taken into a new container. This is because there will be large particles that did not grinded properly and we don't want it to affect the particle size distribution of the sedimentation. Grinding with milling machine or ball grinder usually need sieving because it will contain chips that missed to be grinded. No sieving is required at the beginning only 5-10 min will separate these large particles. The uniform solution after gentle stirring is left undisturbed for 2-6 hours then pure bentonite will be obtained. In purification of Khulays bentonite sometimes multi stage separation was used whenever it appears to be required. The supreme solution is separated and the bottom solution was removed to another container and water is added to decrease the concentration even more. After 2 to 3 stages there will be no bentonite suspension, and the plastic container will contain only clear water at the top and solid non soluble impurities at the bottom. A simple flow chart of the designed process is shown in **Fig.20.**

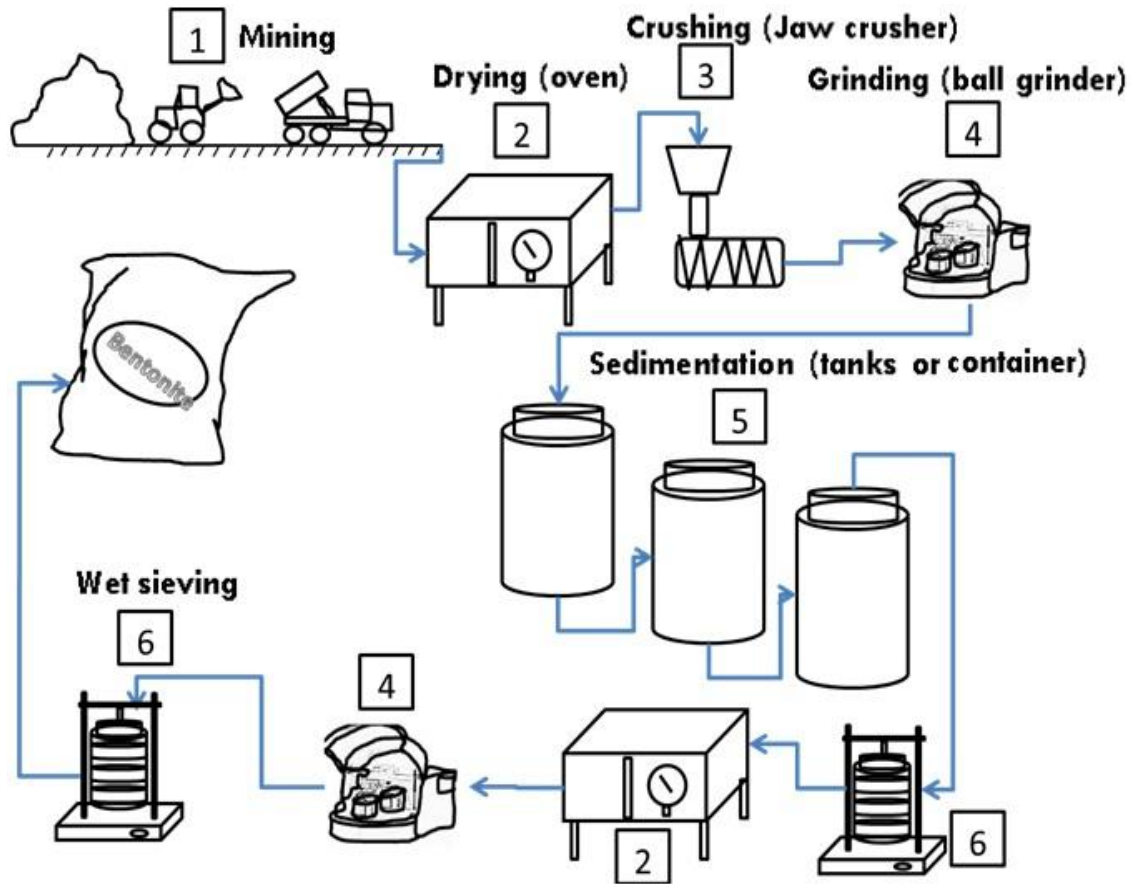


Figure 20 Sedimentation/Sieving method flowchart

## 5.2 Bentonite Characterization

### 5.2.1 Standard Bentonite Characterization

Standard drilling grade bentonite was used as a reference of comparison. Commercial bentonite collected from a local supplier. It represents the typical bentonite used in drilling operations in Saudi Arabia. The commercial bentonite was subjected to all API quality check. The results were set as the benchmark for the local bentonite. The

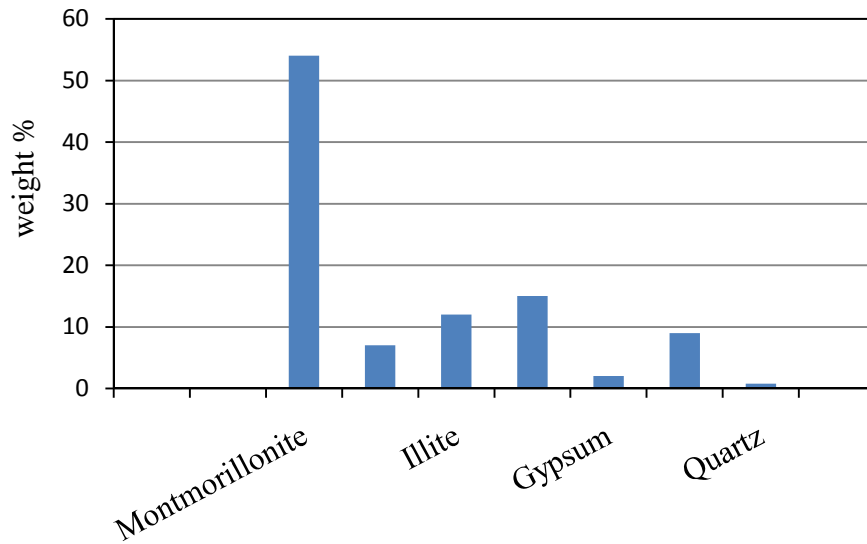
comparison parameters were chemical composition, physical characteristics, particle size, stability, water loss, and rheological properties.

Chemical analysis, X-ray diffraction XRD, XRF and surface area analyzer of scanning electron microscope(SEM) imaging were used for bentonite characterization. The local bentonite was physically and chemically characterized before and after processing and compared with the standard bentonite. XRD analysis listed in **Table.7** and **Fig.21** showed that standard bentonite consists mainly of montmorillonite, which formulate 54% of the bentonite, with other minerals and clay types. Muscovite, illite, cristobalite and feldspar shared considerable amount between 7% to 15%, while quartz and pyrite which usually considered as impurities only exist in a small amounts by 0.8% and 0.1%.

**Table 7 XRD for Standard Bentonite**

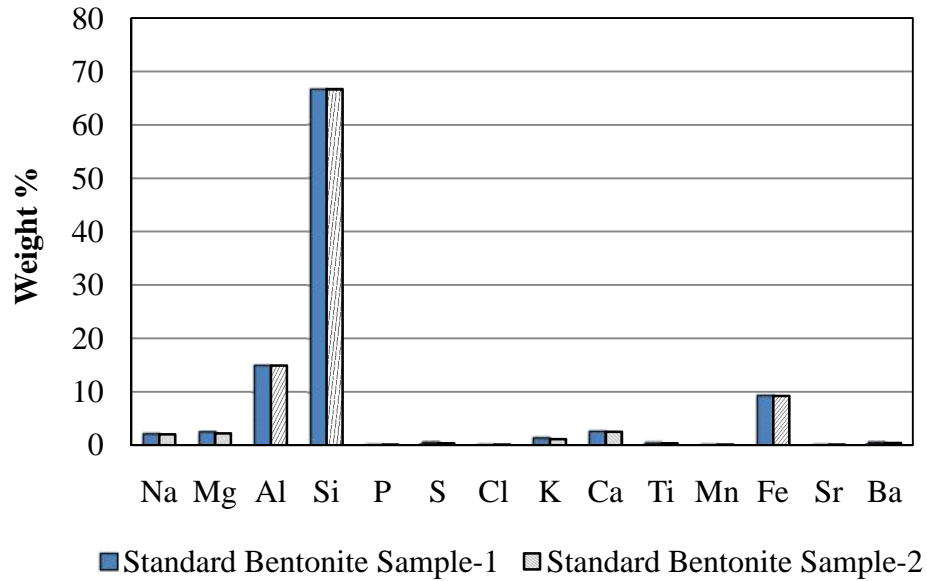
<b>Mineral Phases</b>	<b>Standard Bentonite</b>
Montmorillonite	54.0
Muscovite	7.0
Illite	12.0
cristobalite	15.0
Gypsum	2.0
K-Feldspar	9.0
Quartz	0.8
Pyrite	0.1





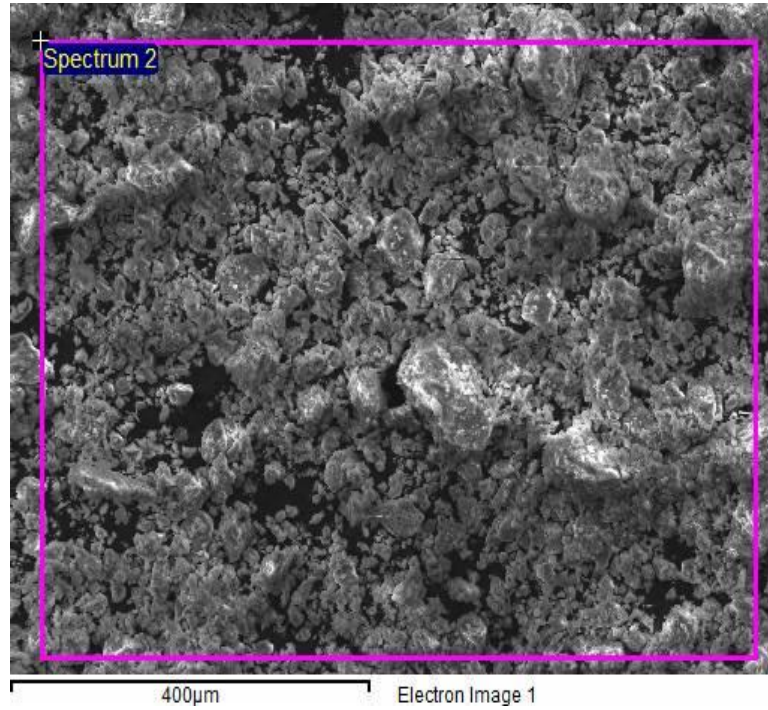
**Figure 21**XRD for Standard Bentonite

The typical montmorillonite formula is  $(\text{Na,Ca})_{0.3} (\text{Al,Mg})_2 \text{Si}_4 \text{O}_{10} (\text{OH})_{2 \cdot n} (\text{H}_2\text{O})$ . The analysis of X-ray fluorescence (XRF) for standard bentonite gave the element and chemical composition of bentonite. The results of analysis showed existence of sodium, magnesium, aluminum, silicon, phosphate, sulphur, chlorine, manganese, and some iron, as showed in **Fig.22**.

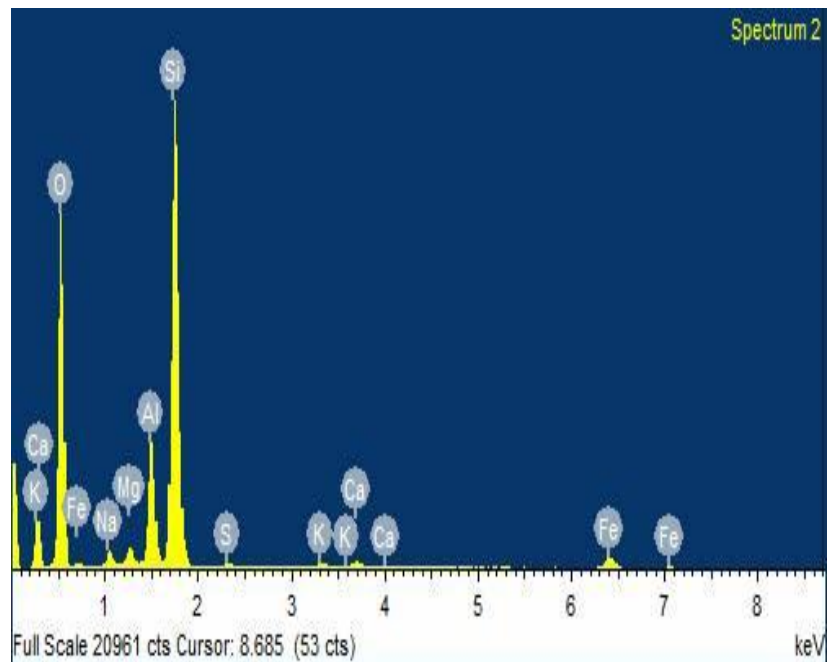


**Figure 22 XRF results for standard bentonite**

Scanning electron microscopy (SEM) was done for the standard bentonite to get more details on the total chemical composition and non-clay minerals of bentonite. **Fig.23** shows the structure of the standard bentonite. SEM image illustrated the typical bentonite structure, layered structure with small bumps and varying degree of roughness. Bentonite used in drilling fluid is required to have a good rheology and swelling characteristic, thus sodium bentonite is preferred. SEM results verified that the standard bentonite is a sodium bentonite, it has high sodium content. Energy dispersive analysis (EDS) gave the chemical elements of standard bentonite. Sodium/Calcium ratio estimated by (EDS) is 2.9 showed in **Fig.24**. Standard bentonite contains oxygen (58.65%), sodium (1.39%), magnesium (0.93%), aluminum (6.83%), silicon (28.59), sulphur (0.24), potassium (0.19%), calcium (0.48%) and iron (2.69%).



**Figure 23 SEM results for Standard Bentonite**



**Figure 24 EDX Analysis for standard bentonite**

Particle size for standard bentonite were measured using two methods, dry sieving and laser scattering particle size analyzer, drilling bentonite must have a uniform particle size distribution, and 96 % of the particles should be less than 75 microns. For sieving analysis, exactly 50 grams of standard bentonite was measured and run through 200 mesh (75  $\mu$ ) and 100 mesh (150  $\mu$ ), sample passed through each sieve were weighted and results are shown in **Table.8**. The standard bentonite was verified to have 94.3 % of the sample below 75  $\mu$ m.

**Table 8 Dry Sieving analysis for standard bentonite**

@ 100 mesh ( 150 $\mu$ )	1.19 gm	2.2 %
@ 200 mesh ( 75 $\mu$ )	1.79 gm	3.5%
@ bottom Tray ( < 75 $\mu$ )	47.1 gm	94.3%
Total	50 gm	100%

For API rheological parameters water loss bentonite base mud was used. Sample of standard bentonite mud was prepared by adding 22.5 gm of bentonite to 350 ml deionized water in mud mixer for 20 min. After 1 day aging the sample was tested in Fann 35 viscometer. The results are shown in **Table.9**. Apparent viscosity was 11.5 cP, plastic viscosity 8 cP and yield point was 7 lb/100ft<sup>2</sup>, filtration was 14.8 ml in 30 minutes and formed 3 mm mud cake.

**Table 9 Standard bentonite, viscometer readings and API filtration results**

Mud composition

Component	lab unit	field unit
Water	350 ml	1 bbl.
Standard Bentonite	22.5 gm	lb

Conditioning	Mixing 20 min, aging 1 day, testing @ 25 o C and atmospheric pressure
--------------	---

MW	8.6
pH	9.5

Dial Speed	Standard
600	23
300	15
200	12
100	8
6	2.5
3	2
10 sec	3
10 min	17

AV	11.5	cP
PV	8	cP
YP	7	Lb/100 ft
API Fluid loss	14.8	ml
Mud cake thickness	3	mm

### **5.2.2 Khulays Bentonite Characterization**

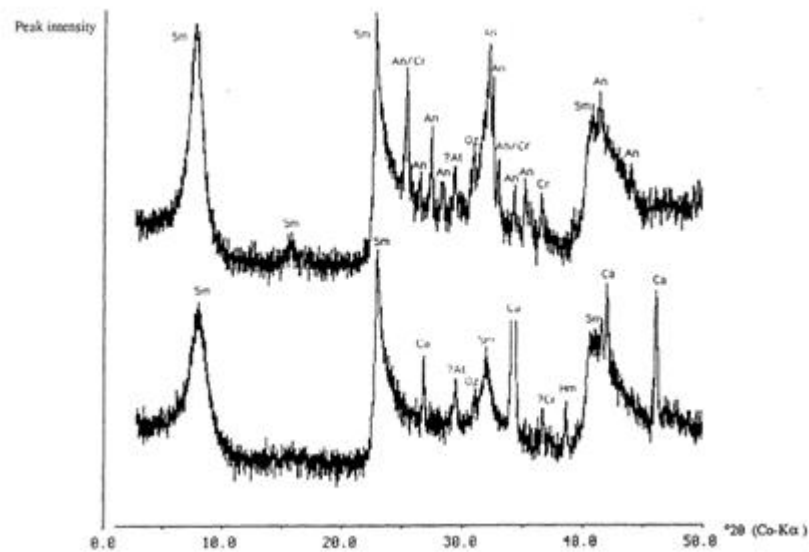
Khulays bentonite was subjected to different chemical and physical characterizations. The collected samples were tested as raw material and also after each purification method. The raw bentonite rock was observed to have two sets of color, first group nominated as bentonite-1, it has gray light color, second group has dark greenish brown color. The structure of the rock has clear uniformed layered beds, and it is not compact but it has loose texture. Some gypsum is observed in some layers with others impurities such as sand. Little black spots of manganese oxide also are clearly observed.

Since bentonite is proven to be derived from volcanic ash it should contain certain minerals, these minerals are known as clay minerals which are used to identify bentonite of montmorillonite clays. X-ray diffraction used to provide information on the mineralogical composition of Khulays bentonite.

#### **5.2.2.1 X-ray Diffraction (XRD)**

**Fig.26** and **Fig.27** shows XRD results of bentonite-1 and bentonite-2, samples tested as powder prepared by grinding the rock after drying it in the oven at temperature around 50-60°C to remove moisture content to less than 10%. The original moisture was around 30%, a 5 kg of bentonite sample after put in the oven for 24 hrs it lost about 20% of its weight as moisture evaporated. The dried samples then crushed and grinded to fine powder to be used in characterization analysis. Bent-1 and bent-2 in XRD test generated almost typical intensity and 2-theta profile. Analysis showed bent-2 has more montmorillonite content and fewer impurities. By comparing the intensity and 2-theta profile to XRD patterns, the peaks height of minerals at the 2-theta degrees showed in

both results were identified as montmorillonite. The XRD patterns of the clay in **Fig.27** clearly show the peak at angle  $26.52^\circ$ . Analysis identified both clay and non-clay content such as quartz, silica and gypsum. Khulays XRD pattern obtained from this test matched the typical raw bentonite XRD pattern described in industrial minerals laboratory manual, which illustrated in **Fig.25**.



**Figure 25** Typical bentonite XRD pattern

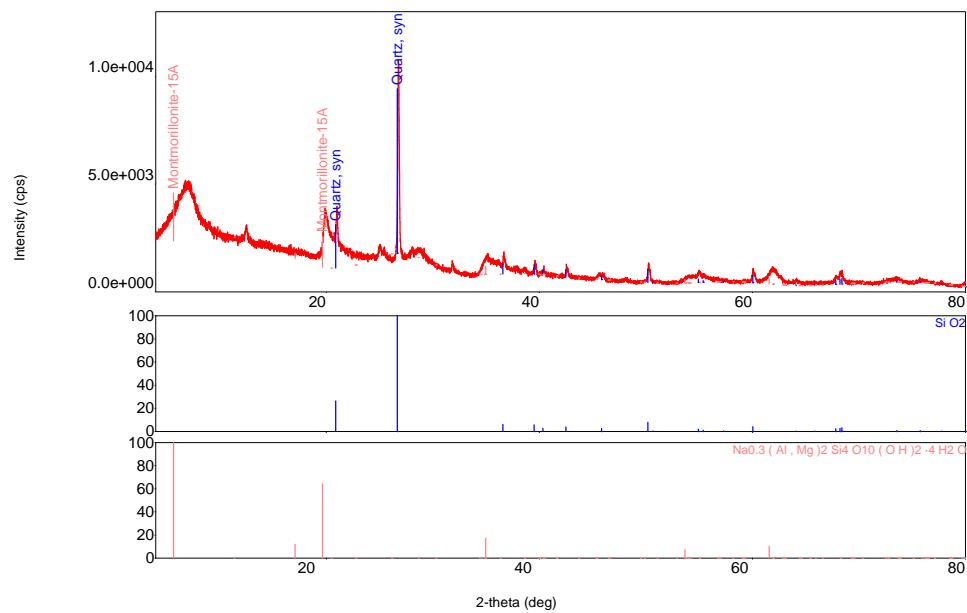


Figure 26 XRD for Bent-1

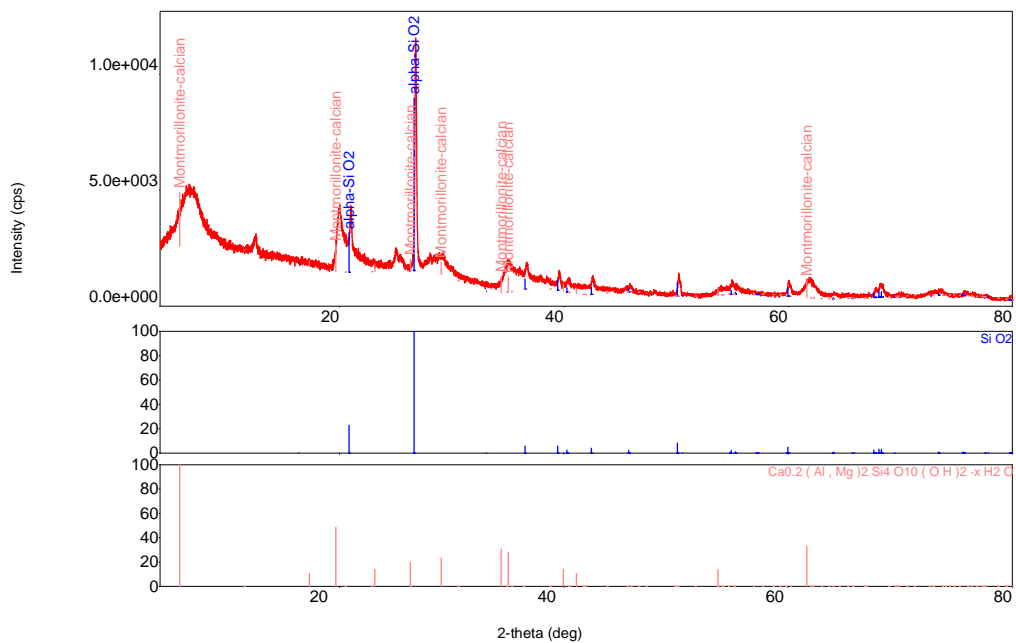


Figure 27 XRD for Bent-2

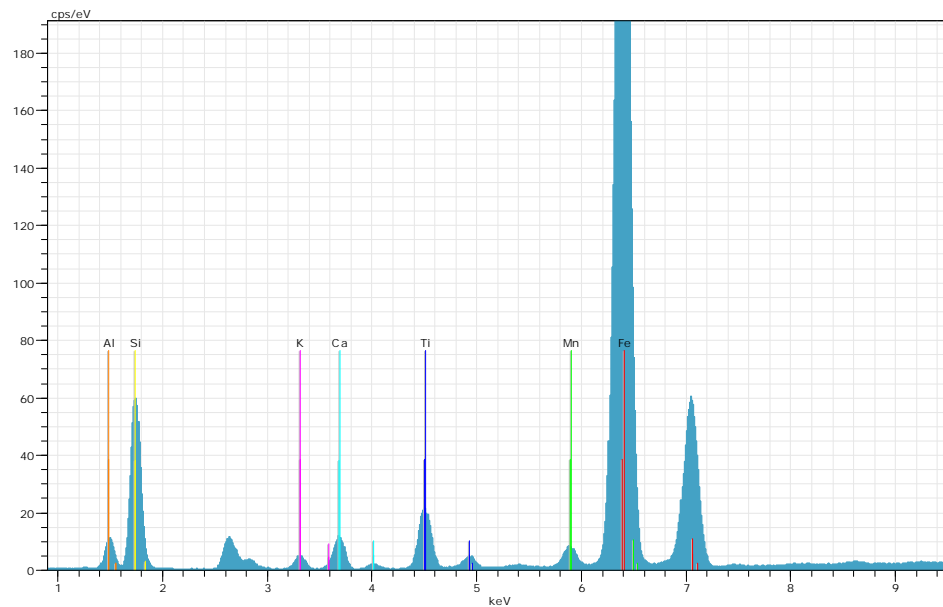


### 5.2.2.2 X-ray Fluorescence (XRF)

X-ray Fluorescence carried out to the raw bentonite and gave the major and trace chemical composition (**Fig.28**).The comparison of all results is shown in **Fig.29** and **Table.10**.The silica content detected was 59% in raw bent-1 and 48.6 in bent-2. Bent-1 value is not far from the standard value of 66.7wt%. The value of silicon in purified bentonite was observed to be decreased to 47wt%; this is mainly due to the quartz that was removed in purification process. XRF also gave an indication that dry sieving was not a good way to purify the bentonite because silicon weight percentage increased, this agreed with the other factors affecting purification process. The quality of the purified bentonite through dry sieving was very poor. Wet sieving without pre-sedimentation also gave high silicon content in sample s1, s2, s3 and s4 the values were 54.35w%, 55.35w%, 56.45w% and 56.26wt%, this is because wet sieving without pre sedimentation will make sure all particles to be washed out by water to the next sieve, both bentonite particles and impurities of quartz, sand and silica. This results support the process that we designed for purifying Khulays bentonite by using Sedimentation/sieving combined method.

The alumina  $\text{Al}_2\text{O}_3$  content detected was 16.09wt% and 15.32wt% for bent-1 and bent-2, these values agree with standard bentonite values of 15wt%, it slightly increased with purification. Iron oxide  $\text{Fe}_2\text{O}_3$  is observed to be much higher in Khulays bentonite compared to the standard bentonite, this explains the brown greenish color of the Khulays bentonite. The value reaches up to 30wt% in raw samples and decreases down to 15wt% in purified samples. This is still high, again this supports that dry sieving or wet sieving without sedimentation is not getting a pure bentonite. While sedimentation/sieving

combined method remove this Iron oxide to less than 3wt%. The raw bentonite contains 2wt% calcium and this value increased by purification to 3wt%, the typical values for calcium bentonite is varying from 1.5wt% to 3wt%. XRF results of Khulays bentonite matches standard bentonite in montmorillonite component such as alumina (15wt %), silica (66wt %) and others small percentages component such as potassium (1.1wt %) and magnesium (2.2wt %).

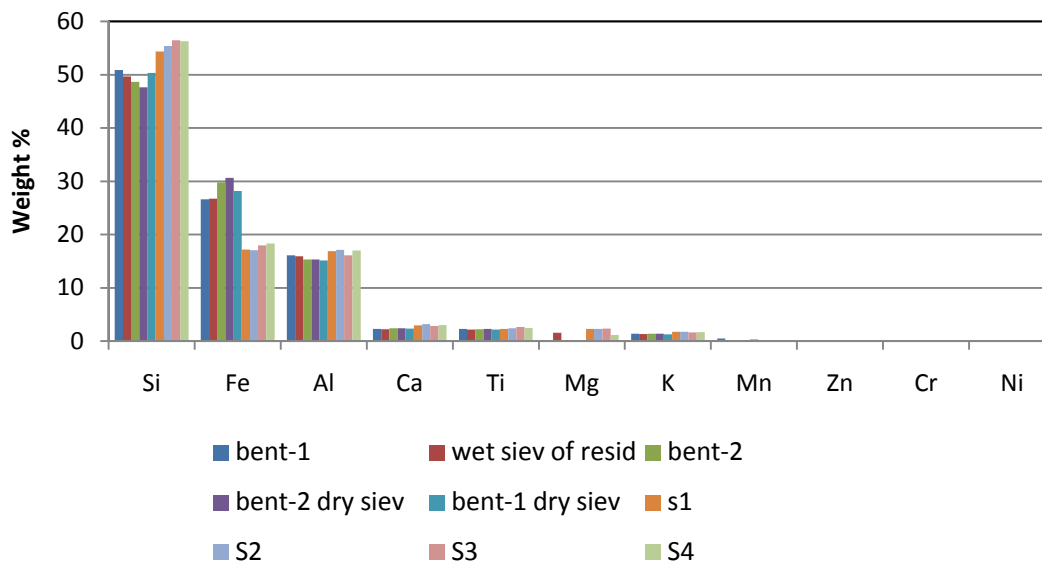


**Figure 28 XRF results for raw bentonite (bent-2)**

**Table 10 XRF results comparison for purification methods**

Element	Standard	Bent-1	Wet sieved	Bent-2	Bent-2 dry sieved	Bent-1 dry sieved	S1	S2	S3	S4
Na	2	0	0	0	0	0	0	0	0	0
Si	66.7	50.89	49.67	48.66	47.61	50.36	54.35	55.35	56.45	56.26
Fe	9.2	26.58	26.72	29.8	30.62	28.16	17.2	17.07	17.93	18.34
Al	14.9	16.09	15.94	15.32	15.34	15.16	16.87	17.09	16.12	16.98
Ca	2.5	2.27	2.24	2.37	2.4	2.34	2.95	3.16	2.85	3.02
Ti	0.3	2.3	2.19	2.24	2.33	2.17	2.29	2.39	2.65	2.49
Mg	2.2	0	1.6	0	0	0	2.26	2.29	2.35	1.16
K	1.1	1.37	1.35	1.37	1.4	1.28	1.74	1.75	1.65	1.66
Mn	0.1	0.48	0.09	0	0.02	0.29	0	0	0	0
Zn		0	0.08	0.08	0.08	0	0.06	0.05	0	0
Cr		0	0.06	0.06	0.07	0	0.05	0	0	0.08
Ni		0	0.04	0.05	0.06	0	0	0	0	0
Ba	0.4	0	0	0	0	0	0	0	0	0

## XRF results comparison



**Figure 29 XRF results comparison for purification methods**

### 5.2.2.3 Scanning Electron Microscopy (SEM)

**Fig.30** shows image by Scanning Electron Microscopy (SEM) to bent-1 sample, and detailed composition was analyzed by Energy Dispersive Spectroscopy (EDS) is described in **Fig.31**. The SEM images for bent-1 sample gave a look at the structure and the shape of the surface of Khulays bentonite. Sample prepared directly from the raw bulk after drying and grinding before purification. Surface of bent-1 appear as random packing particles, it does not have a uniform structure as the standard bentonite shown in **Fig.23**; due to existing impurities in the raw bentonite. The surface has bentonite structure features of wavy layered structure but with high degree of roughness and dull spots. SEM image comparison of bent-1, bent-2 and the standard bentonite clearly shows that bent-2 has more uniform structure and clear wavy layered pattern (**Fig.32**). Both bent-1 and bent-2 has no quoting surface unlike the standard bentonite which appears to be covered a smooth quoting material. This is because usually standard bentonite is not containing only pure bentonite, but it's being enhanced by polymers and some extenders.

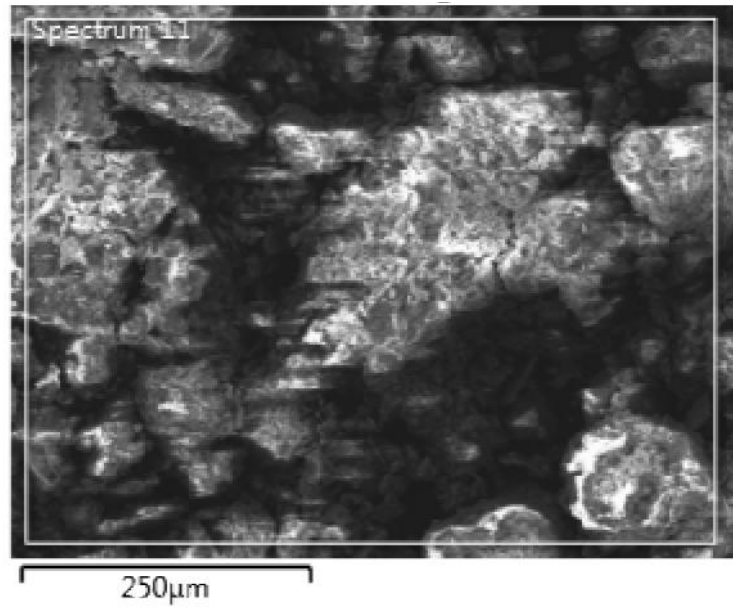


Figure 30 SEM image for Bent-1

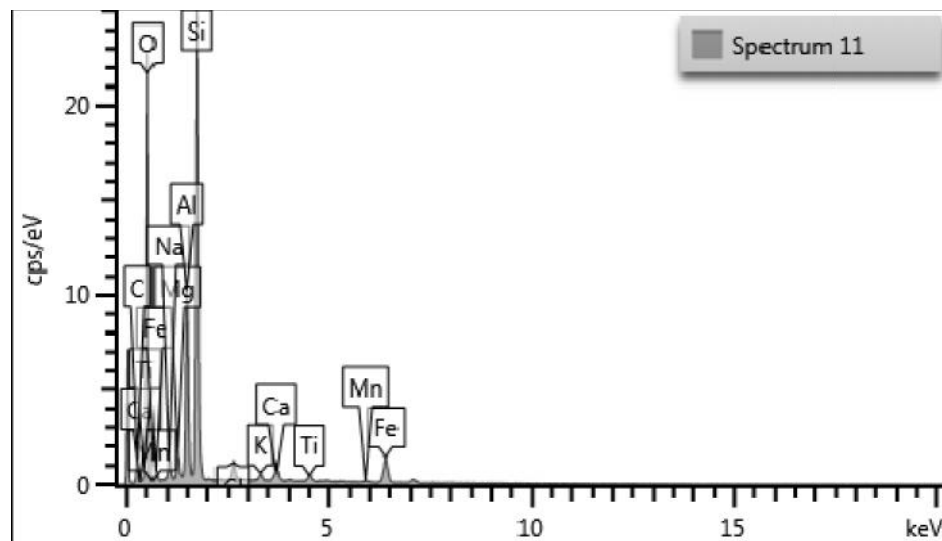


Figure 31 EDX spectrum for Bent-1

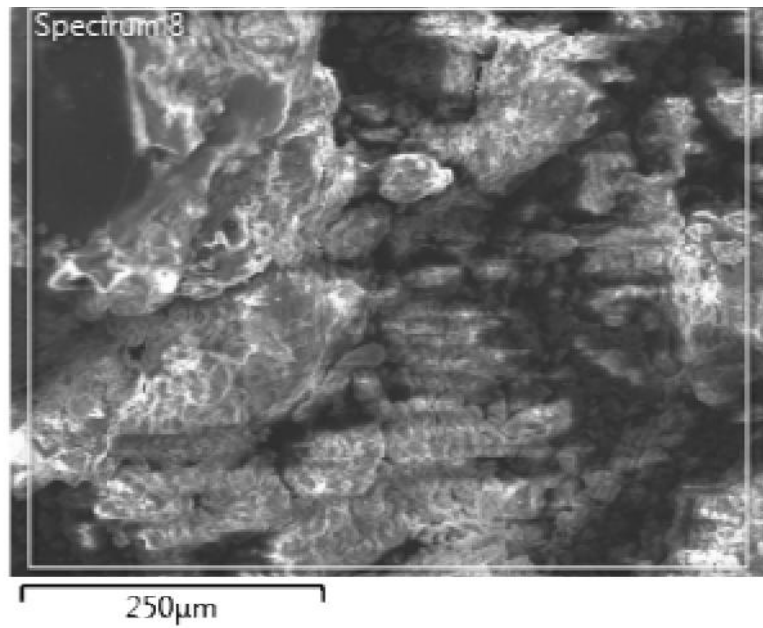


Figure 32 SEM image for Bent-2

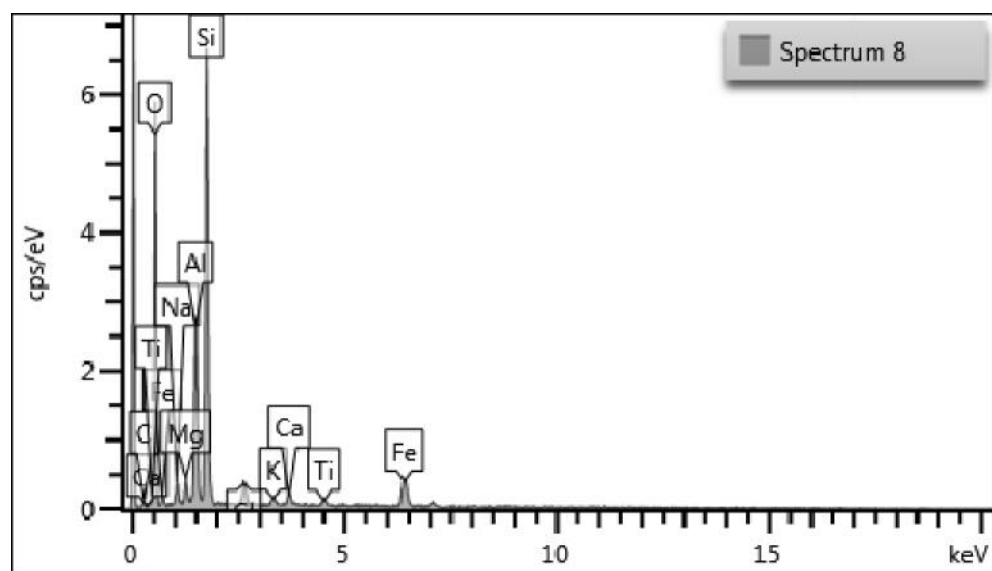


Figure 33 EDX spectrum for Bent-2

**Table.11** shows a comparison to the EDS results for standard bentonite, raw bentonite and processed bentonite with different purification methods. Standard bentonite has a sodium calcium ratio of 2.89 which is sufficient to have a good swelling and rheological properties and qualify it to be used in drilling fluid. Raw Khulays bentonite has sodium/calcium ratio of 1.97 and 1.78 for bent-1 and bent-2. The results show that Khulays bentonite is having most of the elements that formulate the standard bentonite within varying range of weight percentage. The weight percentage of these elements, Oxygen (55.4%-57.6%), silicon (24.28.9), aluminum (6.83%-9.26%), magnesium (0.93%-1.55) and potassium (0.19%-0.48%), all in the same range same as the standard bentonite. The difference between the standard and Khulays bentonite is the sodium\calcium ratio, beside the share of small percentage of some impurities elements such as iron, manganese, chlore, phosphor and cupper. Manganese detected in the raw bentonite, it shares 0.7%-0.9% by weight, which is considerable amount in Khulays bentonite compared to the standard bentonite. This is verifying what has been observed before grinding there was black spots indicate magnesium oxide. This manganese can improve gel strength significantly and it's favorable in drilling fluids.

**Table 11.a SEM Results for samples purified through different methods**

<b>Element</b>	<b>Standard</b>	<b>BN-6</b>	<b>BN-7</b>	<b>SED-1</b>	<b>S-1</b>	<b>S-2</b>	<b>S-3</b>	<b>S-4</b>
<b>O</b>	58.65	54.04	51.19	54.52	55.46	52.37	51.82	57.27
<b>Na</b>	1.39	2.35	1.73	0.75	1.13	0.8	0.88	0.79
<b>Mg</b>	0.93	1.58	1.46	1.4	1.55	1.5	1.63	1.41
<b>Al</b>	6.83	8.87	9.59	9.16	9.26	9.8	10.74	8.7
<b>Si</b>	28.9	24.3	25.1	24.08	24.57	27.32	26.17	22.97
<b>P</b>	-	1.25	-	-	-	-	-	-
<b>Cl</b>	-	0.98	1.22	0.14	1.25	0.45	0.74	0.43
<b>K</b>	0.19	-	0.54	0.58	0.48	0.55	0.43	1.68
<b>Ca</b>	0.48	0.8	0.91	0.82	0.95	0.92	0.96	0.65
<b>Ti</b>		0.53	0.83	0.79	0.7	0.78	0.88	1.42
<b>Fe</b>	2.69	5.59	6.64	5.53	4.65	5.52	5.76	5.63
<b>Cu</b>	-	-	0.89	1.28	-	-	-	-
<b>Zn</b>	-	-	0.88	0.94	-	-	-	-
<b>S</b>	0.15	-	-	-	-	-	-	-
<b>Total</b>	100%	100%	100%		100%	100%	100%	101%
<b>Na/Ca ratio</b>	2.896	2.95	1.891	0.915	1.182	0.873	0.913	1.209



**Table 12.b SEM Results for samples purified through different methods**

<b>Element</b>	<b>Standard</b>	<b>A-1</b>	<b>B-3</b>	<b>B-7</b>	<b>W-1</b>	<b>R-1</b>
<b>O</b>	58.65	50.62	51.3	51.19	50.88	51.67
<b>Na</b>	1.39	1.73	1.69	1.73	1.77	1.79
<b>Mg</b>	0.93	1.43	1.44	1.46	1.36	1.36
<b>Al</b>	6.83	9.44	9.56	9.59	9.49	9.46
<b>Si</b>	28.9	26.24	25.28	25.1	25.31	24.82
<b>P</b>	-	-	-	-	-	-
<b>Cl</b>	-	1.51	1.4	1.22	1.6	1.39
<b>K</b>	0.19	0.61	0.6	0.54	0.59	0.56
<b>Ca</b>	0.48	0.98	0.95	0.91	0.89	0.87
<b>Ti</b>	-	0.93	0.81	0.83	0.89	0.79
<b>Fe</b>	2.69	6.51	6.97	6.64	2.83	6.13
<b>Cu</b>	-	-	-	0.74	6.57	0.82
<b>Zn</b>	-	-	-	0.88	-	0.71
<b>S</b>	0.15	-	-	-	-	-
<b>Total</b>	100%	100%	100%	100%	100%	100%
<b>Na/Ca ratio</b>	2.896	1.756	1.789	1.891	1.978	2.043

**Fig.34** compares some of the purification methods resulting in obtaining pure bentonite after removing impurities and non-clay materials. Bent-1 has less iron and non-clay elements but contain high copper percentage (6.5%). Bent-2 is containing high iron (6.9%) compared to the standard bentonite (2.6%), but it's rich in calcium, silicon, aluminum and magnesium. **Fig.36** shows purification method efficiency in removing of iron from raw bentonite, wet sieving using 38 micron sieve get the iron percentage below 5% and bent-1 has 2.83 % of iron, about the same as the standard bentonite.

**Fig.35** compares the sodium/calcium ratio of the standard bentonite against Khulays bentonite as raw and purified. The standard bentonite has the highest Na/Ca ratio of 2.89. Khulays bentonite as raw is having Na/Ca ratio less than 2. Based on these results shown in **Fig.35**, the purification methods cannot increase Na/Ca ratio to a significant level, because purification process remove the impurities which is exists in small percentage and doesn't affect Na/Ca ratio which is depend on the amount of sodium and calcium ion in the bentonite. Khulays bentonite is proven to be calcium bentonite and to increase sodium calcium ratio a treatment should be introduced to increase sodium calcium ratio.

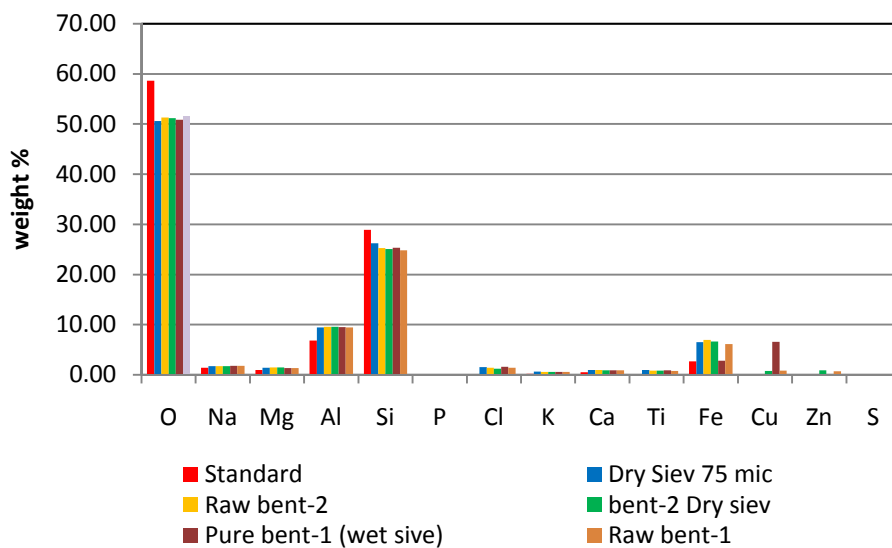


Figure 34 Chemical Composition of Different Purifications Methods

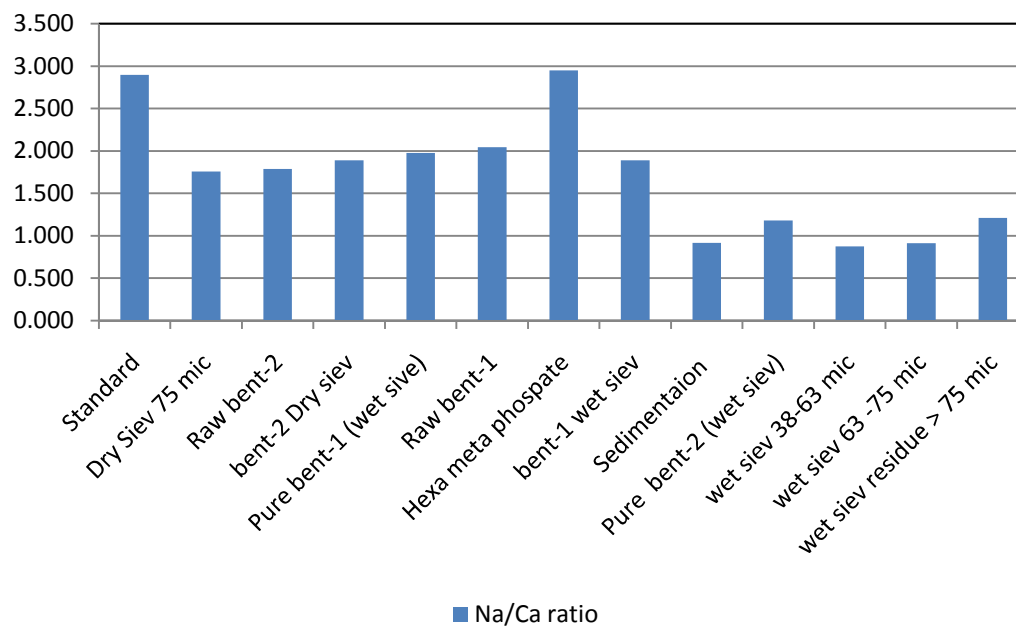


Figure 35 Na/Ca ratio for different purifications methods

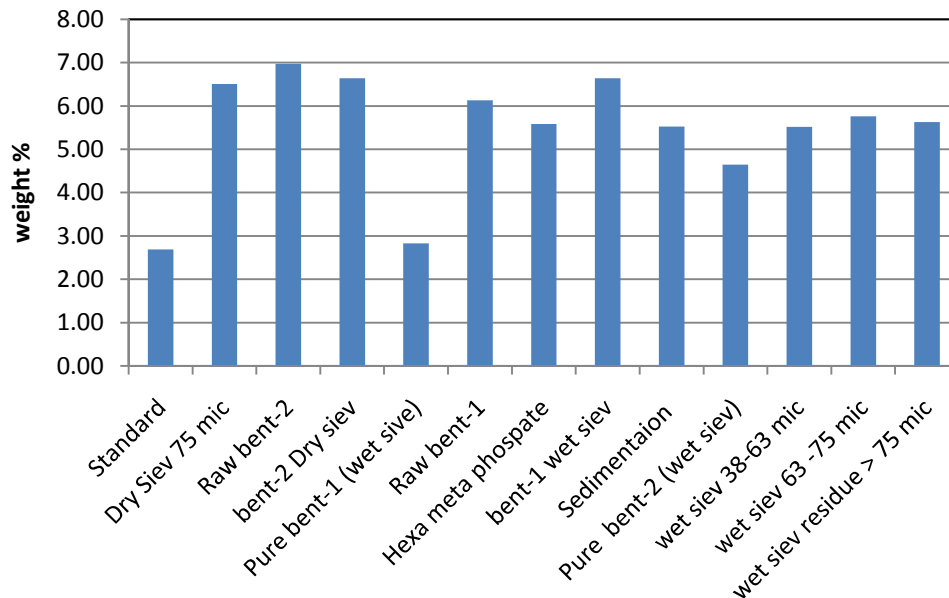


Figure 36 Iron content for different purifications methods

### 5.2.3 Suspension Stability and Zeta potential (ZP)

Stability of pure bentonite was evaluated using zeta potential (ZP) which is a measurement of particle charges. Zeta potential measuring gives an analytical understanding of the interaction forces and separation mechanism of bentonite particles in water suspension. **Fig.37** compares raw untreated bentonite against the standard bentonite, even at high pH the local untreated bentonite is having a low ZP values and this results agrees with the quick phase separation occurred to the sedimentation samples. **Fig.38** shows ZP results for standard bentonite at different pH prepared through two different purification methods. Activation of local bentonite with soda ash and heating/stirring treatment improved the dispersion characteristic of the local bentonite; this is clearly verified by the measuring of zeta potential for the treated samples. The results on **Fig.39** that ZP has moved to more negative values compared to the raw

untreated bentonite. This will provide repulsive charges on the surface of the bentonite particles which will ensure that particles will remain dispersed.

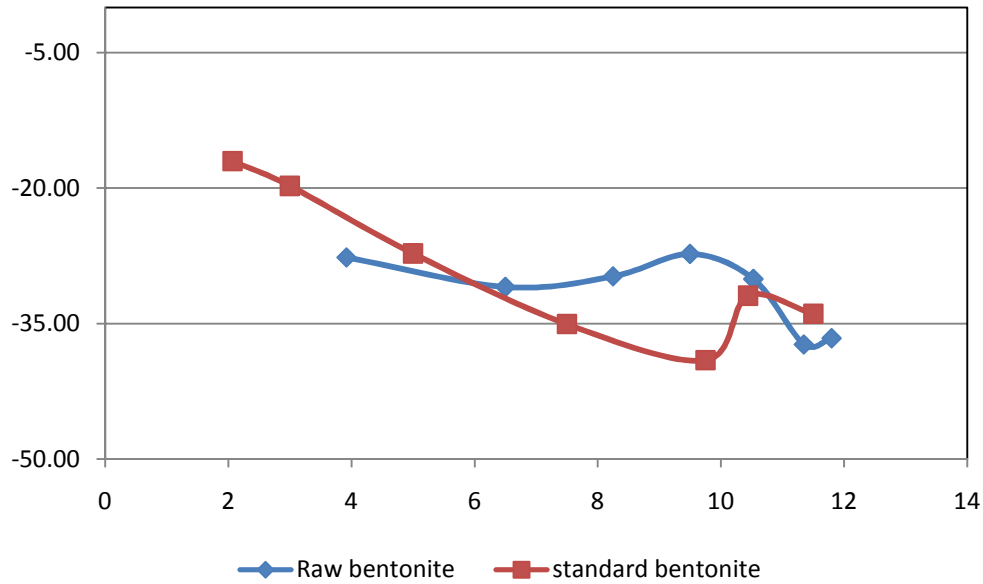


Figure 37 ZP for standard and Raw Bentonite

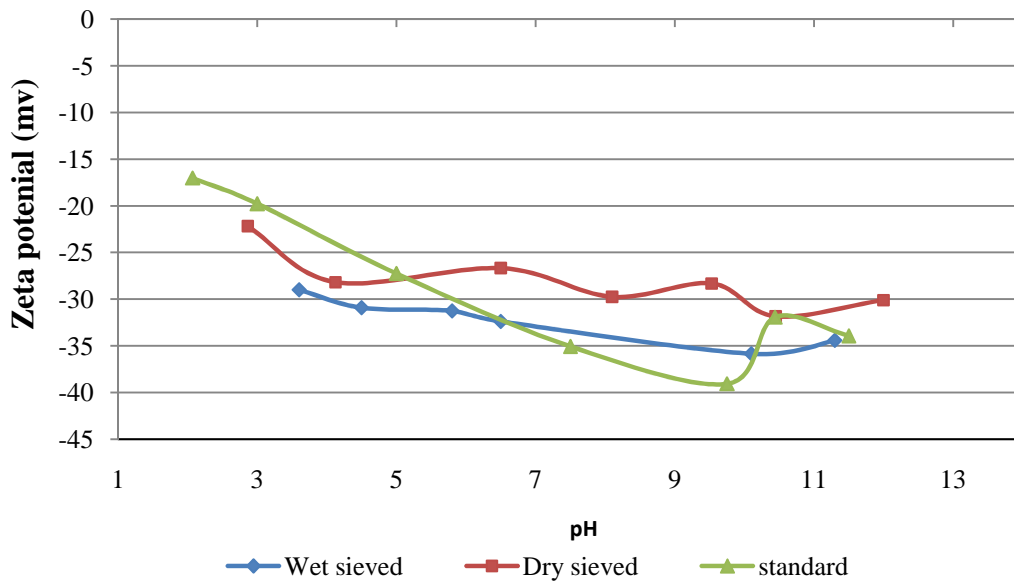


Figure 38 Zeta Potential for standard and purified local bentonite

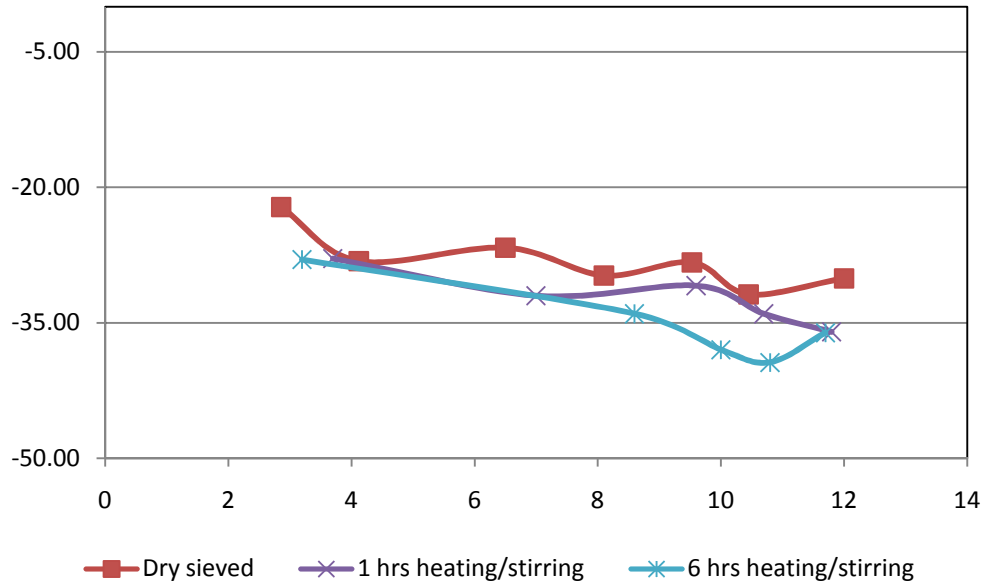
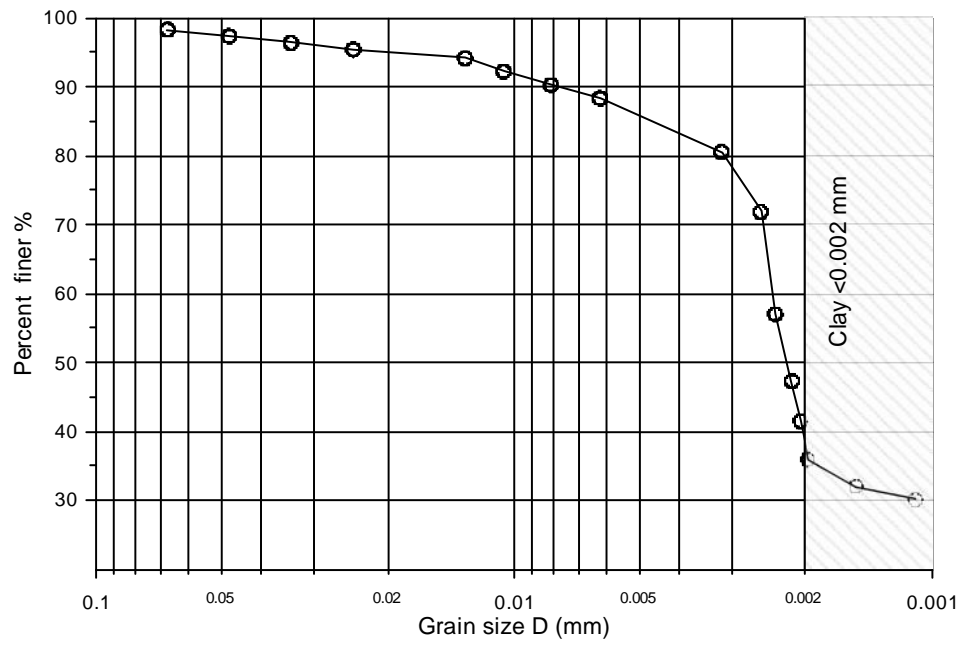
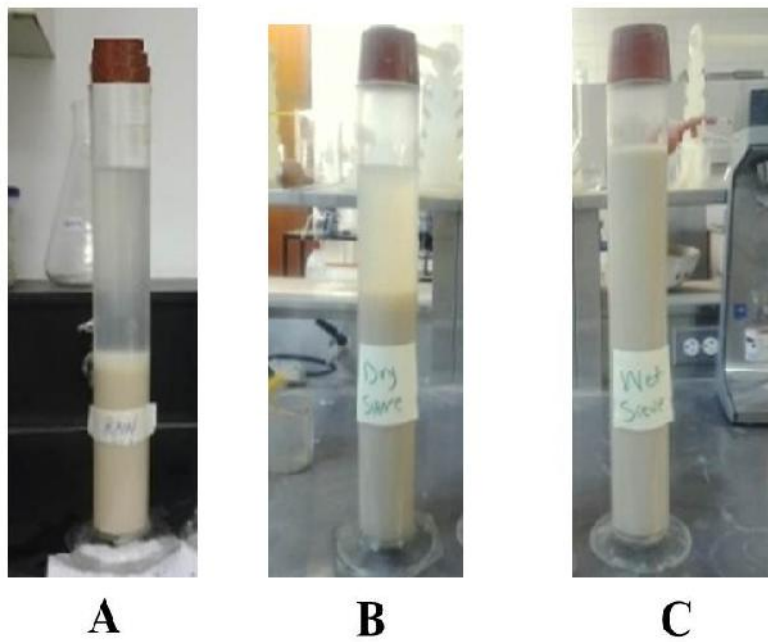


Figure 39 ZP for treated bentonite

Hydrometer analysis was used to estimate clay content in raw bentonite. The particle size distribution is measured for the small size particles of clay. The content of clay, sand and silt was estimated and shown in **Fig.40**. When the sample is mixed with water the particles settled at different velocities, depending on their shape, size, weight and viscosity of water. The velocity of soil particles can be expressed by Stokes' law. Since the silt has a higher grain size than clay (clay is ~ 4 microns while sand has 0.45-2 mm), the silt will fall to the bottom before the clay does, leaving only silt and clay particles in suspension. Then silt settles and leaves only clay in the suspension. Dry sieved sample A-1 contains 35% as clay, while wet sieved samples are almost 100 % clay.



**Figure 40 Hydrometer Analysis for sample A-1**



**Figure 41 Settling rate of hydrometer test, A-raw, B-dry sieving, C-wet sieving**

Raw and purified bentonite samples were used to prepare 1000 ml solution by mixing 50 gm of bentonite into 125 ml of 4% solution of deflocculating agent such as sodium hexametaphosphate then the mixture was completed to 1000 ml with water, and left for 72 hrs. in graduated cylinder, the pH was kept as original value 9.5. **Fig.41** shows the settlement and migration of the interface based on different settling rate. **Fig.41A.** shows that raw bentonite exhibited the higher settling rate and bentonite obtained by wet sieving purification method **Fig.41C** was stable for 72 hrs. Hydrometer analysis was used to estimate clay content, dry sieved sample **Fig.41B** contains 35% as clay, while wet sieved samples **Fig.3C** are almost 100 % clay.

### 5.3. Ca-Bentonite Treatment

The local bentonite is a Ca-bentonite and soda ash treatment was used to increase Na/Ca ratio to improve swelling and dispersion. The raw bentonite was purified using Sedimentation/sieving combined method after screening of different purification methods. All non-clay impurities rather than montmorillonite was removed, and a pure ca-bentonite was obtained. **Table.13** shows the sodium/calcium ratio of from SEM results, of raw and purified bentonite compared to the standard bentonite. It is clear that local bentonite is Ca-bentonite and need to be Na-activated by increasing Na/Ca ratio to improve swelling and reach the required rheological behavior. Two batches of local bentonite, bent-1 and bent-2 were treated with soda ash and aged for different times. Below are detailed results of soda ash treatment and bentonite reaction with it at different doses and aging time.



**Table 13 Sodium/Calcium ratio for standard and local bentonite**

<b>Sample</b>	<b>Sodium %</b>	<b>Calcium %</b>	<b>Na/Ca Ratio</b>	<b>Comments</b>
Standard Bentonite (SEM)	1.39	0.48	2.90	From SEM results Standard Drilling Grade bentonite
Raw (A1)	1.73	0.98	1.77	Batch_2 Dry sieved 200 mesh (75 micron)
Purified (W1)	1.79	0.89	2.01	Batch_2 wet sieved 200 mesh (75 micron)

### **5.3.1 Bent-1 Sodium Activation**

Treatment with soda ash was carried out by taking 22.5 gm of bent-1 to prepare 350 ml mud with density of 8.6, the slurry concentration is 6 wt%. Soda ash was added to the water before that and stirred in high speed mixer for 5 minutes. Bentonite suspension sample tested in dial speed viscometer to measure the rheological parameters. About 8 samples was prepared using the same procedures by adding soda ash to the water and mix for 5 minutes, then adding raw pure bentonite and mix for 20 minutes. The samples represent different doses of soda ash as follow: (0.0 gm, 0.5 gm, 1.5gm, 2.5 gm, 3 gm, 4 gm, 5 gm, and 7 gm). Apparent and plastic viscosity increased with soda ash addition and a peak was observed when 3 gm of soda ash were used as shown in **Fig.42**. **Fig.43** shows the comparison between treated raw bentonite (bent-1) and standard bentonite. It's obvious that standard bentonite is way above bent-1 properties, in Apparent viscosity and

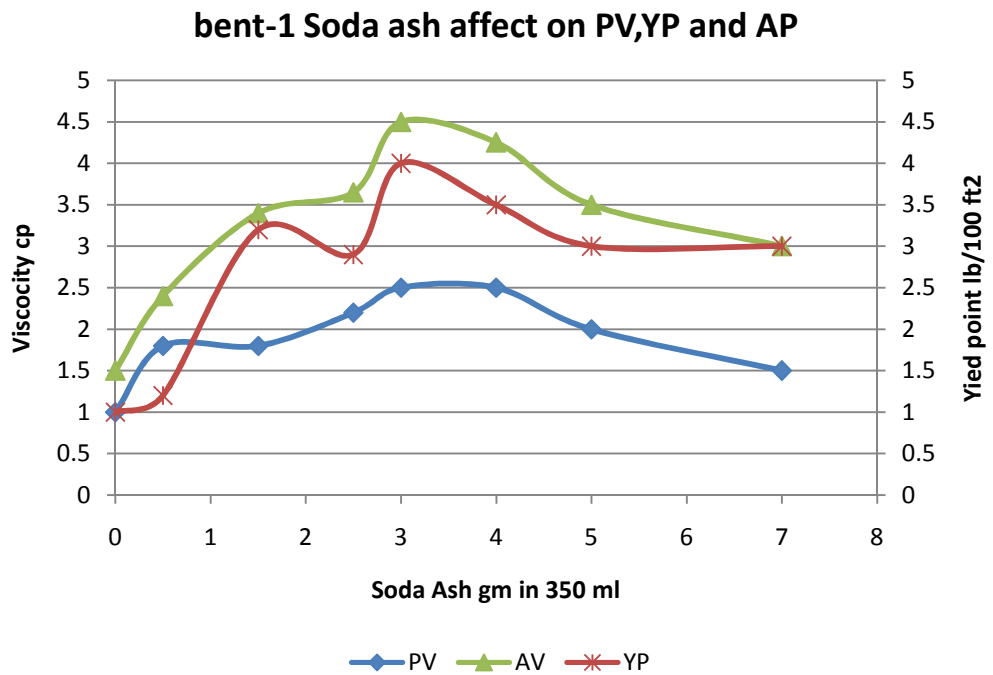


Figure 42 bent-1 Soda ash affect on PV,YP and AP

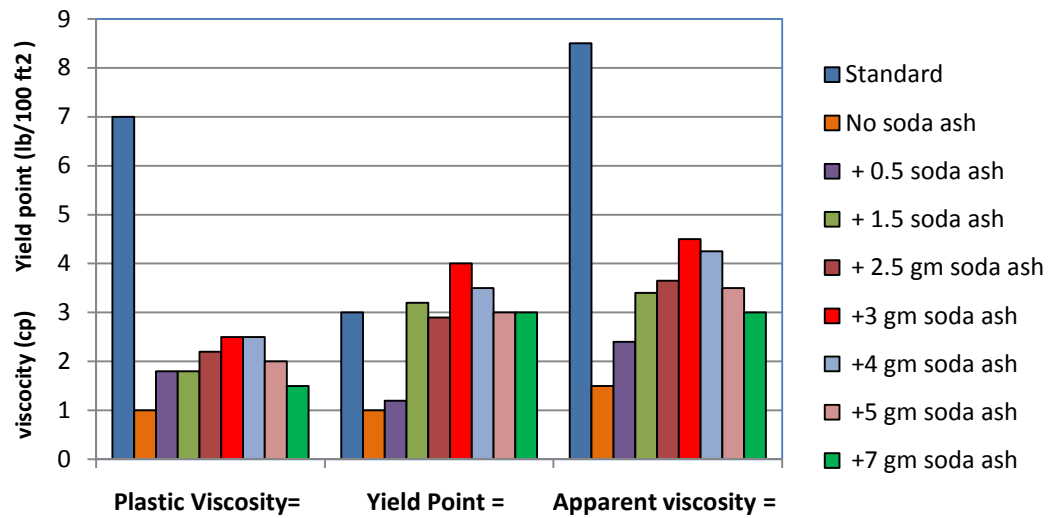


Figure 43 bent-1 Rheology after soda ash activation

plastic viscosity ( $AV = 4.5cP$  to  $8.5cP$ ,  $PV = 2cP$  to  $7cP$ ), while yield point appears to be higher is bent-1 than standard bentonite ( $PV = 4$  to  $1 \text{ lb}/100 \text{ ft}^2$ ).

Since the higher peak was at 3 gm soda ash, further treatment was carried out to this point. Aging of bentonite suspension allow dehydration and improve bentonite properties in water. Aging affect at bent-1 with 1 gm soda ash was evaluated, the reading of dial speed viscometers are shown in **Table.14**. bent-1 showed some improvement in 600 and 300 reading, its illustrated in **Fig.45**, so apparent viscosity and plastic viscosity slightly increased with aging time through 6 days (shown in **Fig.44**).

**Table 14 Aging affect at bent-1 with 1 gm soda ash, viscometer readings**

Dial Speed	Standard-1	Standard-2	NO Soda	Soda ash treated			
				No Aging	1 Day aging	3 Days aging	6 Days aging
600	17	23	3	7	9	12	12
300	10	15	2	5	6.5	9	9.5
200	8	12	1.3	4	5	7.9	7
100	5	8	0.9	3	4	6.9	6.5
6	1	2.5	0.2	2.3	3	4	4
3	0.8	2	0.1	2.1	3	4	4
10 sec		3		3.5		7	
10 min		17		5		12	

PV	7	8	1	2	2.5	3	2.5
YP	3	7	1	3	4	6	7
Av	8.5	11.5	1.5	3.5	4.5	6	6

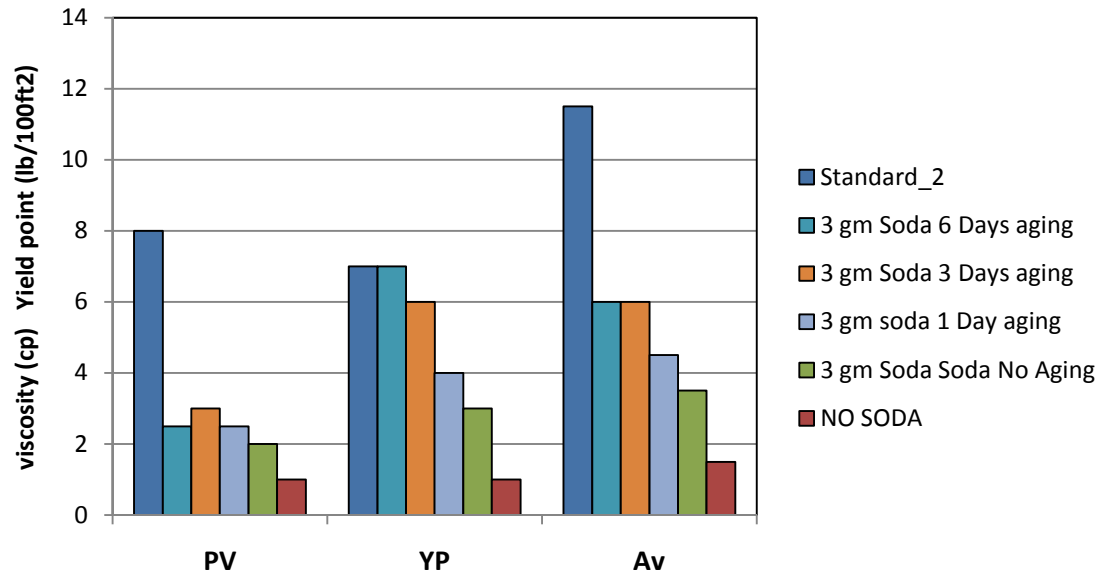


Figure 44 Affect of Aging and addition of 3 gm Soda Ash

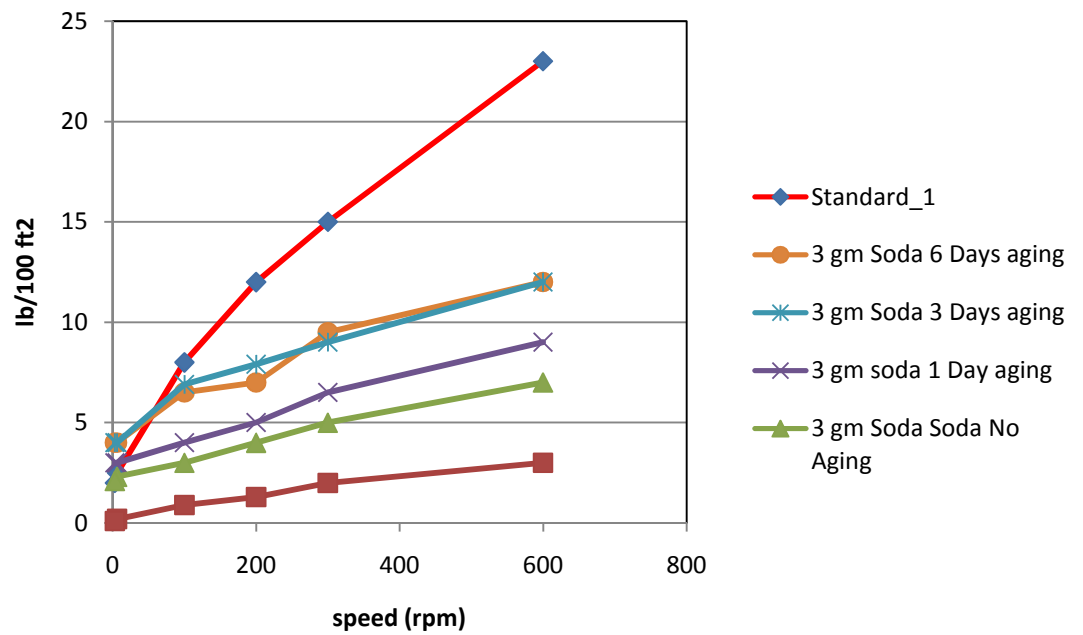


Figure 45 Viscometer dial speed reading after soda ash addition

### 5.3.2 Bent-2Sodium Activation

The second batch of purified bentonite (bent-2) was treated with soda ash in different doses, as follow: (0.5gm, 1gm, 2gm and 3gm). For 6wt% bentonite suspension, the properties improved and viscosity increased with soda ash up to 1 gm. added to 22.5 gm. of bentonite, then decreased again with soda ash addition, which indicated that maximum amount of soda to completely replace the calcium in raw bentonite is 1 gm and any excessive addition will reverse the affect, it's shown in **Fig.46**. Apparent viscosity and yield point exhibit an increasing and decreasing with a peak at 1 gm, but plastic viscosity doesn't change much.

**Tables.15-17** show the effect of aging and soda ash treatment, the sample aged for 1 day and 2 days and compared to the fresh samples, although aging allowed for hydration of bentonite, it showed only little improvement with time, and even decrease in very long aging time. For 0.5 gm of soda ash, the 600 and 300 reading of dial speed viscometer increased from 7 and 4.5 to 8 and 5. For 1 gm of soda ash, the 600 and 300 reading of dial speed viscometer increased from 9 and 6.5 to 10 and 7.

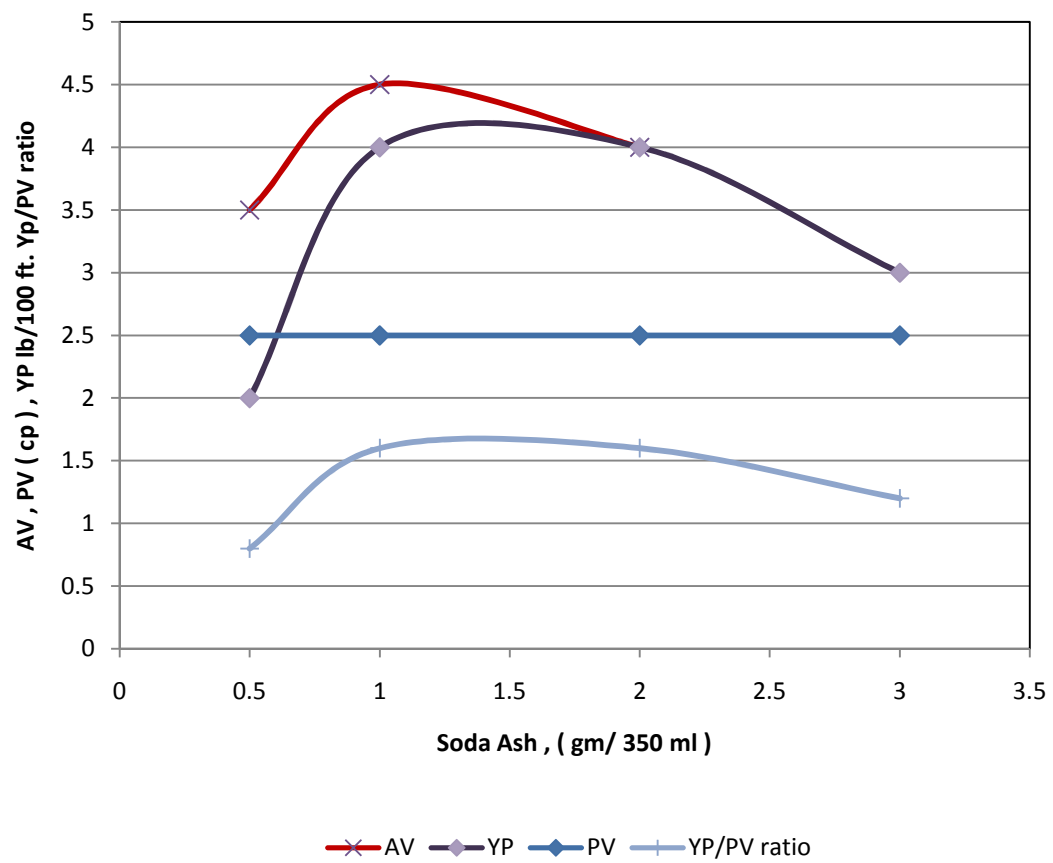


Figure 46 Bent-2, optimum amount of soda ash activation

**Table 15 Table 15 effect of aging and soda ash amount on viscometer readings (No aging)**

Dial Speed	Standard_1	Standard_2	no Aging			
			0.5 gm soda	1 gm soda	2 gm soda	3 gm soda
600	17	23	7	9	9	8
300	10	15	4.5	6.5	6.5	5.5
200	8	12	3.5	5	5	4.5
100	5	8	2.5	4	4	3
6	1	2.5	1.5	3	3	2.5
3	0.8	2	1.5	3	3	2.5
10 sec		3	1.5	3.5		3
10 min		17	3.5	5		6

PV	7	8	2.5	2.5	2.5	2.5
YP	3	7	2	4	4	3
Av	8.5	11.5	3.5	4.5	4.5	4

**Table 16 Table 15 effect of aging and soda ash amount on viscometer readings (1 Day aging)**

<b>Dial Speed</b>	<b>1 Day Aging</b>			
	<b>0.5 gm soda</b>	<b>1 gm soda</b>	<b>2 gm soda</b>	<b>3gm soda</b>
<b>600</b>	8	10	9	8
<b>300</b>	5	7	6	5
<b>200</b>	4	6	5	4
<b>100</b>	3.2	5	4	3
<b>6</b>	2	4	4	2.5
<b>3</b>	2.5	5	4	3
<b>10 sec</b>	2.5	4.5	4	4
<b>10 min</b>	6	10	10	7

<b>PV</b>	3	3	3	3
<b>YP</b>	2	4	3	2
<b>Av</b>	4	5	4.5	4



**Table 17 Table 15 effect of aging and soda ash amount on viscometer readings (2 Days aging)**

Dial Speed	<b>2 Days aging</b>			
	0.5 gm soda	1 gm soda	2 gm soda	3 gm soda
600	4.5	5	10	13
300	2.5	2.8	7	8
200	2	2	5.5	6
100	1.2	1.5	4	4.5
6	0.5	0.5	3	2.5
3	0.5	0.5	2.5	2.5

PV	2	2.2	3	5
YP	0.5	0.6	4	3
Av	2.25	2.5	5	6.5

The rheological properties alteration due to soda ash treatment and aging are shown in **Table.18** and **Fig.47-50**. bent-2 at 6wt% suspension in water when treated with soda ash and left in room temperature for 1 day gave the best results. The red line in **Fig.49** shows that apparent viscosity of 4.5 cP which is higher than the same sample when tested as fresh. Yield point also showed some increasing up to 4 lb/100 ft<sup>2</sup>, but plastic viscosity was not affected by aging.

Since bent-2 with only 1 gm of soda ash is giving the highest possible rheological properties, bent-2 was selected for all further treatments. Bent-1 cost at least 3 gm of soda ash to attain the same properties. The maximum possible values obtained by soda ash treatment and aging was not sufficient for drilling fluid application. Drilling fluid requires at least 15 cP plastic viscosities, with minimum value of 30 dial speed reading. The yield point to plastic viscosity ratio should be at least 3. Bent-2 in the best cases gave apparent viscosity of 12 cP at 24 dial speed reading, plastic viscosity 6 cP and very high yield point of 12 lb/100ft<sup>2</sup>.

**Table 18 Rheological properties after aging and soda ash treatment**

Soda (gm)	PV			YP			AP		
	no aging	1 D	2 D	no aging	1 D	2 D	no aging	1 D	2 D
0.5	2.5	3	2	2	4	4	3.5	4.5	4.5
1	2.5	3	2.2	4	4	3	4.5	4.5	4
2	2.5	3	3	4	3	2	4	4	5
3	2.5	3	5	3	2	4			

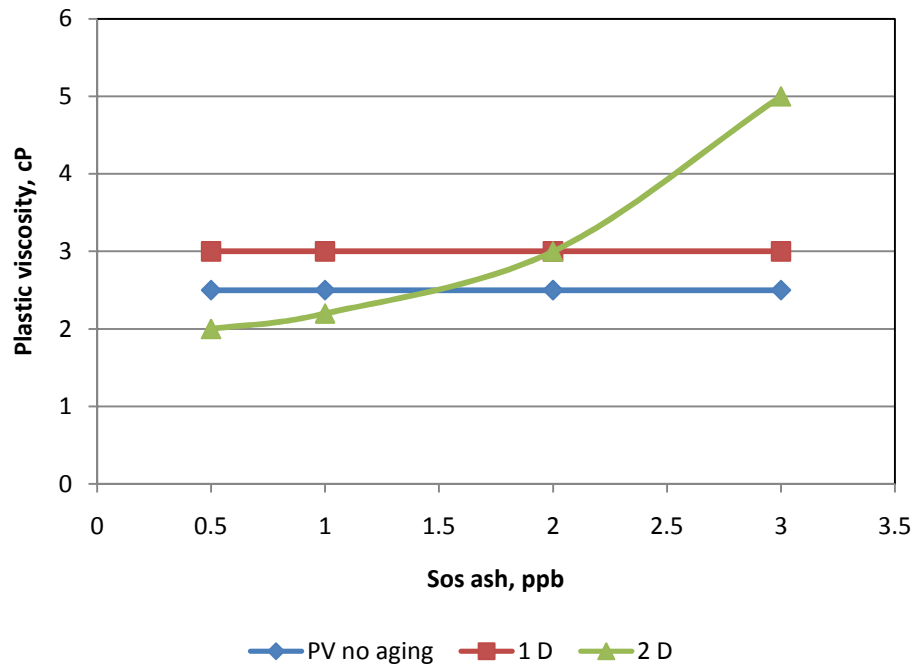


Figure 47 Effect of aging on plastic viscosity

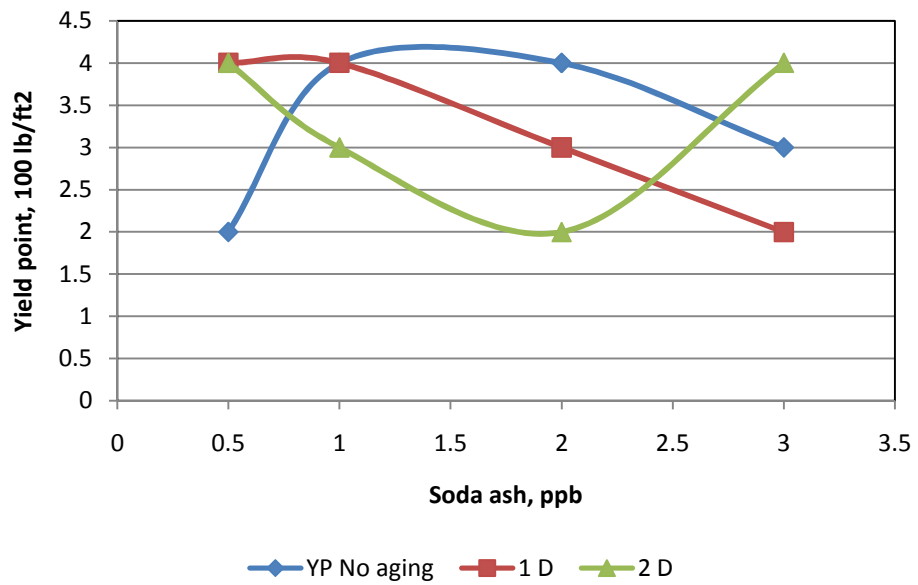


Figure 48 Effect of aging on Yield point

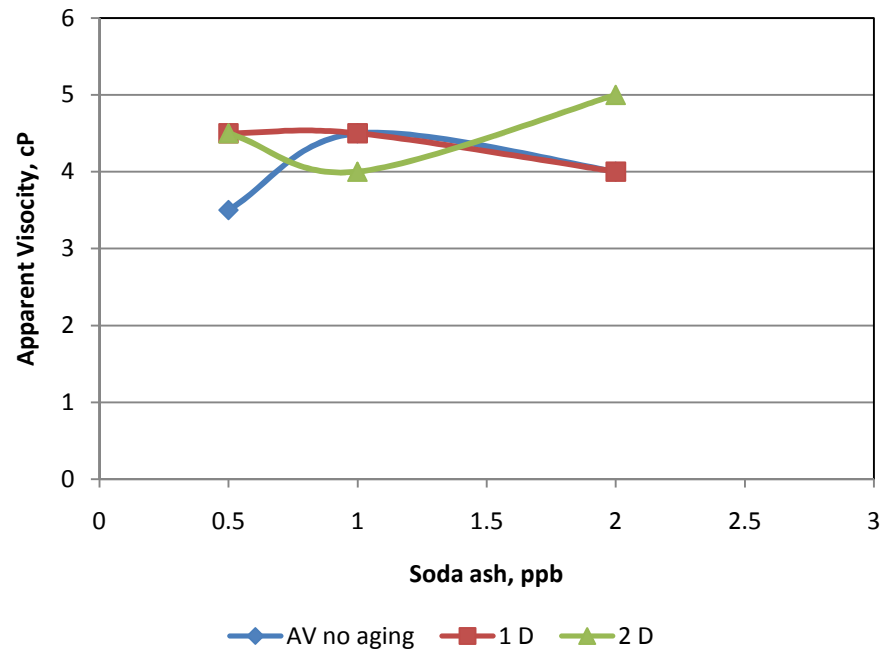


Figure 49 Effect of aging on apparent viscosity

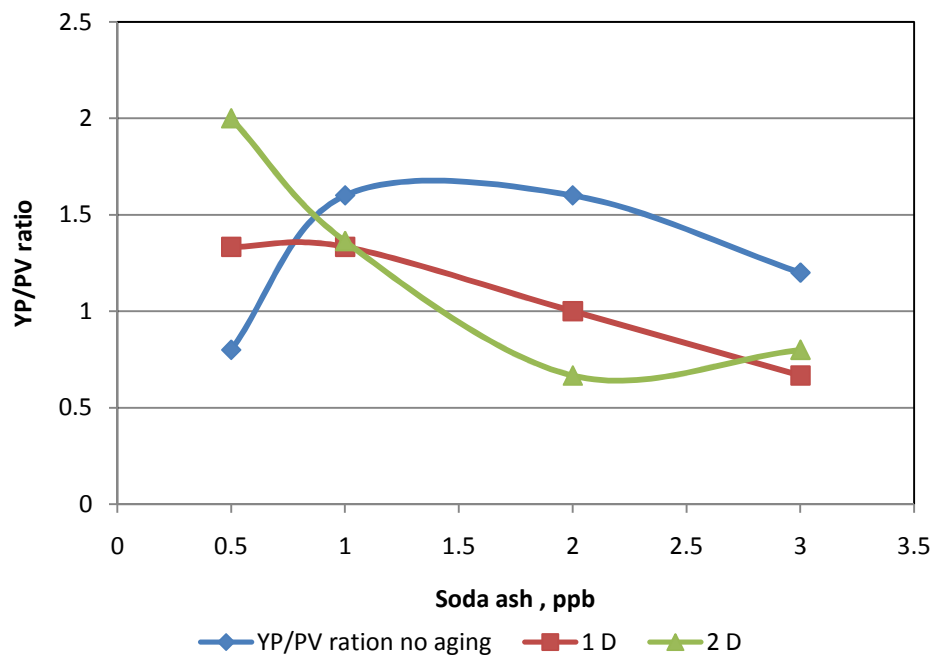


Figure 50 Effect of aging on YP/PV Ratio

### 5.3.3 Hot Stirring Treatment

The common activation method is carried out by adding 2 to 3% of soda ash to water. Then, bentonite was added and mixed to allow ion exchange of sodium and calcium. This will increase Na/Ca ratio and change Ca-bentonite into Na-bentonite which has an increased swelling capacity and better rheological properties. In treating bent-1 soda ash consumption was 3 gm added to 22.5 gm of the bentonite, that's value is more than 13wt% soda ash, which is much higher than the feasible amount of soda ash being used in the industry. Bent-1 samples exhibited better reaction with soda ash and consumed only 1 gm which is about 4 wt% which is accepted as reasonable cost wise treatment. Apparent and plastic viscosity increased and a peak was observed when 1 gm and 3 gm of soda ash were used as shown in **Figs.42** and **Fig.64**.

Based on these results it was proven that for Khulays bentonite, adding soda ash only does not give the required swelling of sodium activated bentonite. In bentonite water suspension preparation, API recommends using hot rolling for 16 hrs to allow for dehydration, and properties of bentonite suspension will be attained after that. The conditions are described by API as 150 °F (65°C) for 16 hrs at a pressure of 120 psi in closed pressurized cell. The API procedure for hot rolling was used but no improvement on rheology was observed. When hot rolling is used with treated bentonite there was little improvement in the viscosity, maximum of 12 cP apparent viscosity. This does not satisfying API specifications. Therefore, we introduced a novel approach to treat Khulays bentonite or any calcium bentonite with soda ash by hot stirring. This method exhibits great enhancement in swelling capacity and rheology.

Samples were prepared by mixing 22.5 gm of bentonite in 350 ml of distilled water as recommended by API specification. Soda ash added to water and mixed for 5 min then 22.5 gm of bentonite was added and mixed for 20 minutes. The samples then treated with the proposed novel method of heating/stirring after soda ash activation. In this method samples were heated at 70°C and stirring for a specific time to enhance the Na-activation and the swelling process. At the end of the hot stirring process, water lost due to evaporation is compensated for by deionized water to maintain the same bentonite concentration in the suspension. Water loss average after 24 hours heating was about 50 to 70 ml.

**Fig.51** shows how heating/stirring method greatly improved rheological properties, the apparent viscosity increased significantly from 8.5 up to 12 when soda ash is used as 3 gm. The amount of soda ash of 3 gm was not the optimum value, even though the viscosity increased after hot stirring treatment. **Fig.52** shows how reading of dial speed viscometer increased through heating/ stirring methods. When the optimum amount of soda ash (1 gm soda ash to 22.5 gm bentonite) is used combined with heating stirring method the viscosity exhibited outstanding improvement from 1 cP to 20 cP. **Fig.53** shows the improvement on apparent viscosity, plastic viscosity with heating time, it shows that zero heating time has the lowest values and values increased when heated to 1 hr and 2 hrs, the maximum values were obtained at 24 hrs heating/stirring. A screening of the optimum time is shown in the following, to define the minimum time required to attain the required swelling capacity.

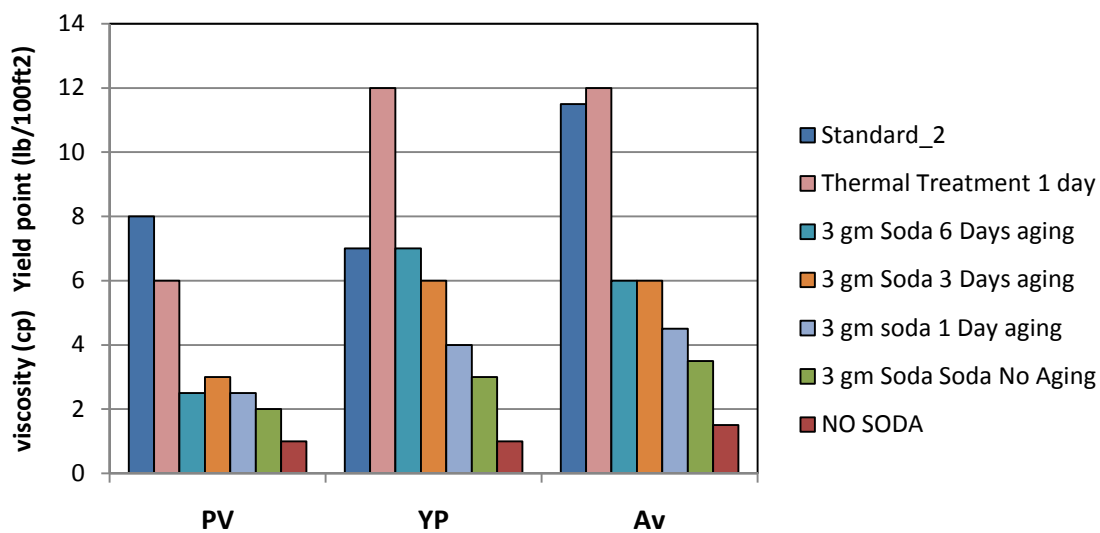


Figure 51 Effect of aging and heating on 3gm Soda ash treatment

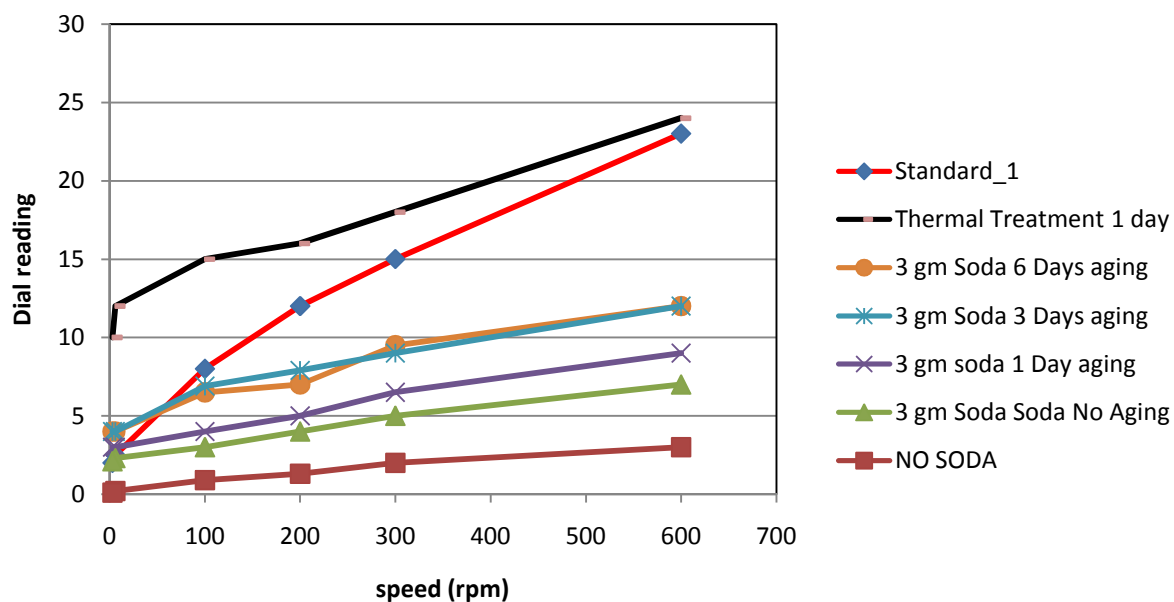


Figure 52 Reading of dial speed viscometer for 3 gm SA heating/ stirring methods

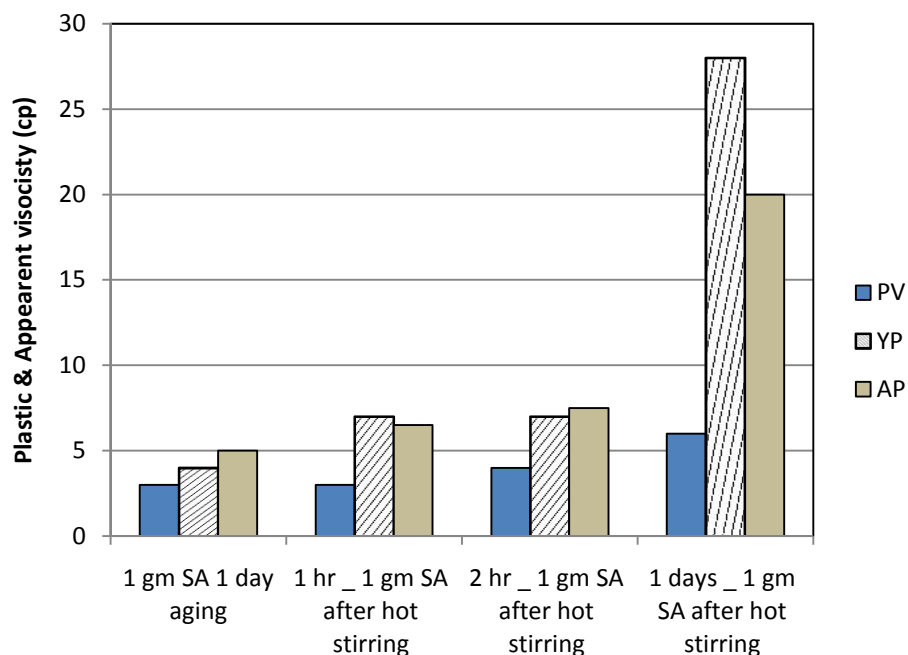


Figure 53 Bent-2 soda ash and heating/stirring treatment

To set the minimum time for the process of treatment, the heating/stirring duration was varied from 1-24 hours. **Fig.54** and **Fig.55** indicate that a minimum of 3 to 6 hrs is needed to achieve the maximum viscosity. Figure 56 and 57 show the viscosity buildup with heating time at low and high shear rate. It indicates that after 6 hrs of heating the viscosity can reach its peak and no further significant increment will take place. Lower heating times could be used to achieve optimum values.



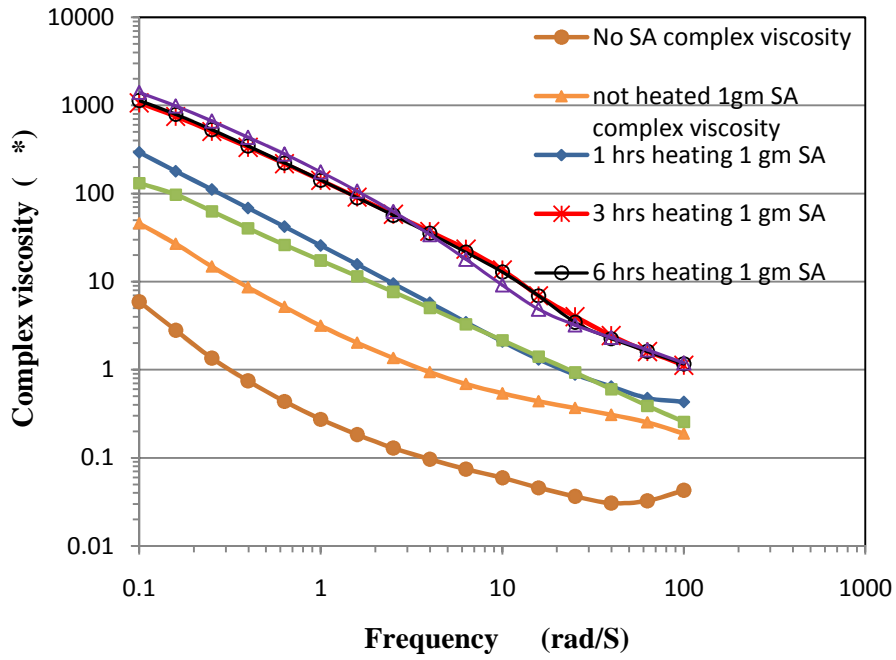


Figure 54 Influence of heating/stirring time on complex viscosity for 1 gm SA sample

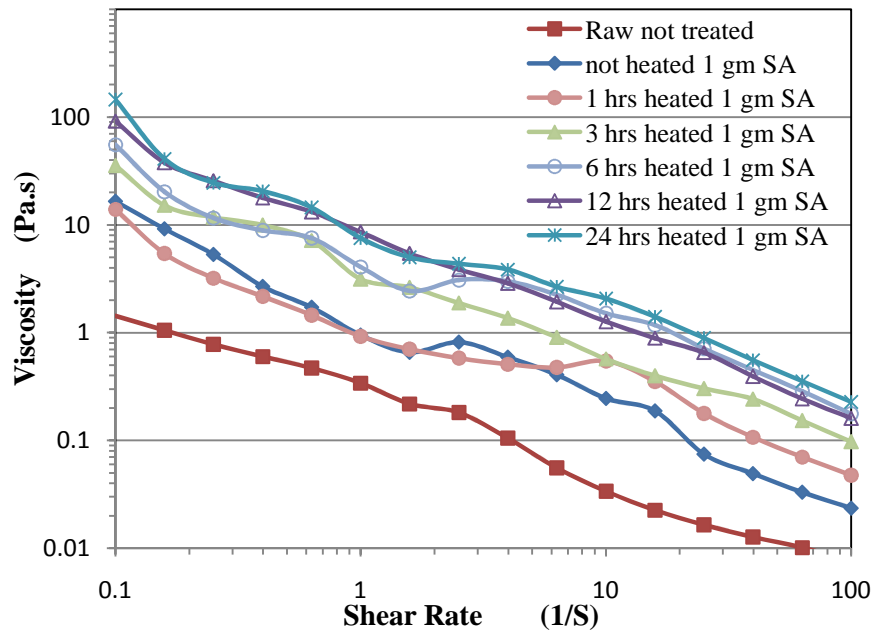
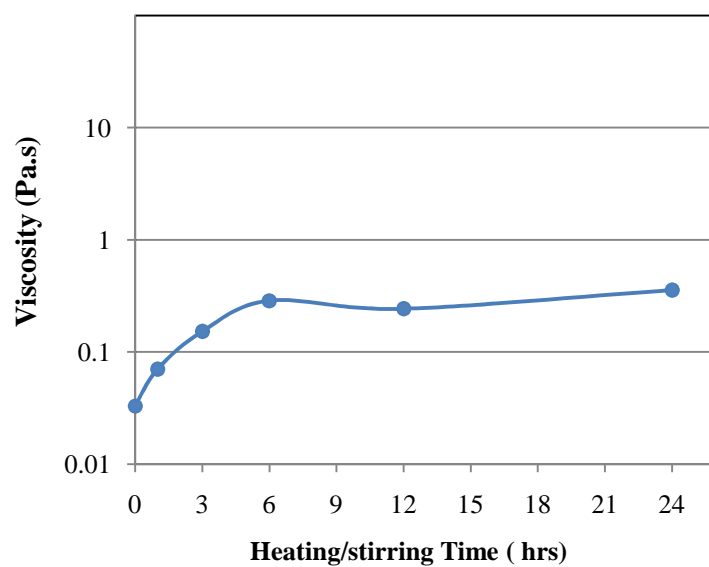
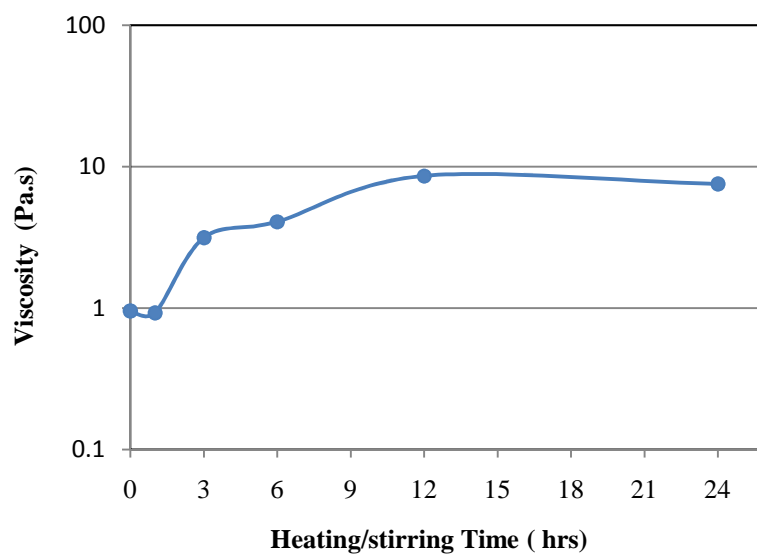


Figure 55 Influence of heating/stirring time on shear viscosity



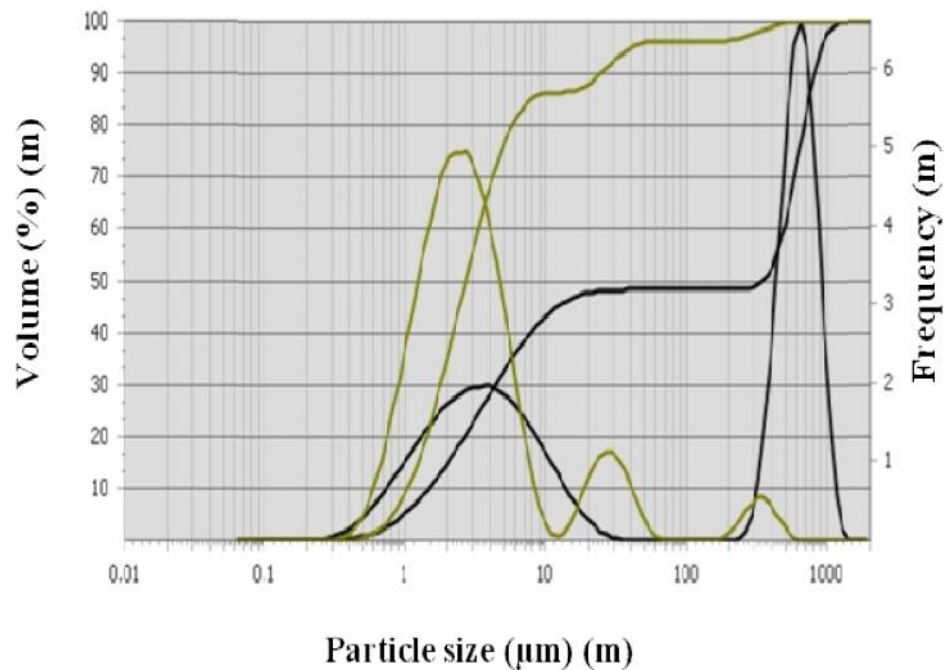
**Figure 56 Influence of heating/stirring on viscosity at high shear rate (64 s-1)**



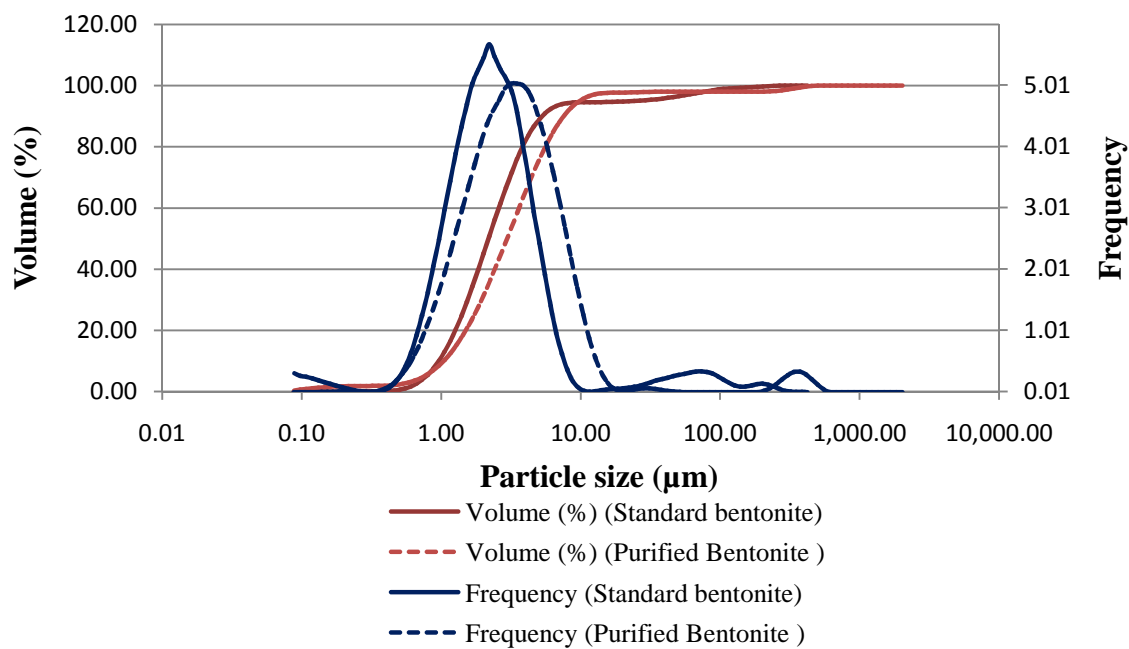
**Figure 57 Influence of heating/stirring on viscosity at low shear rate (1 s-1)**

## 5.4 Particle Size Analysis (PSA)

Frisch Type Particles Size analyzer (PSA) was used to obtain the full particle size distribution. The results showed that the raw bentonite has wide range of particle sizes from 0.8  $\mu\text{m}$  up to 2000  $\mu\text{m}$ . Particle size distribution for raw bentonite is shown in **Fig.58** with comparison against the standard bentonite. After purification, the larger particles were removed. **Fig.59** shows the particle size distribution for wet sieved samples (400 mesh) after sedimentation. The majority of impurities were removed and the size distribution has matched that of standard bentonite. **Fig.59** and **Table.19** shows the comparison between local bentonite after purification and the standard bentonite.



**Figure 58**Size distribution of bent-1 raw samples (not sieved)



**Figure 59 Particles size distribution for purified bentonite**

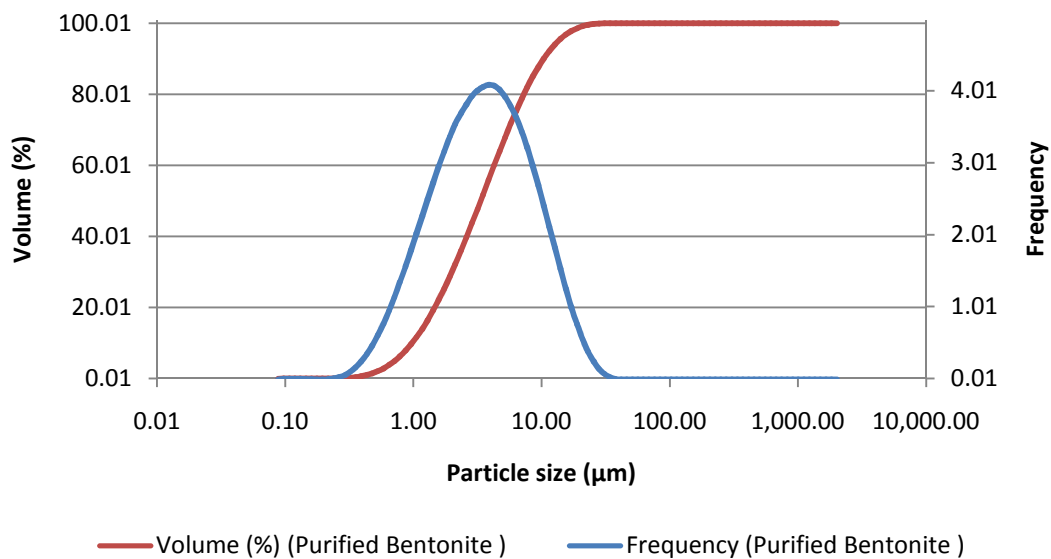
**Table 19 Comparison of particle size of raw and standard bentonite**

Vol [%]	Local Particle size [ $\mu\text{m}$ ]	Standard Particle Size [ $\mu\text{m}$ ]
5	0.8	0.7
10	1.1	1
25	1.6	1.6
50	2.8	2.8
75	5	4.7
90	29.8	7
95	57.4	8.9
99	395.3	18.3

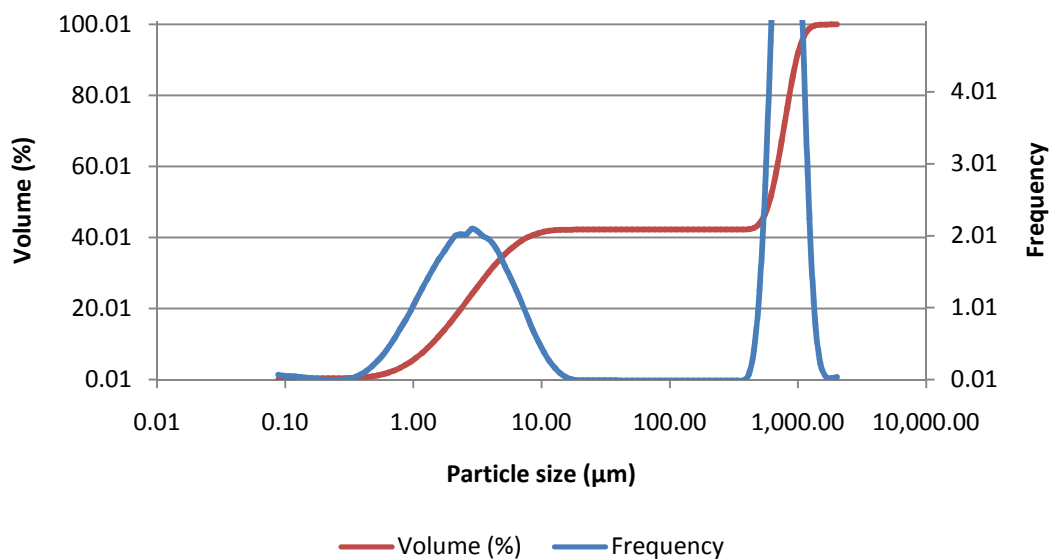
Montmorillonite particles are less than 2  $\mu\text{m}$ , API and OCMA specifications recommended that drilling grade bentonite should have particle size less than 75  $\mu\text{m}$  and the residue that retained by 200 mesh is less than 4% when 50 gm of bentonite is sieved. The standard bentonite samples passed 47.1 gm out of 50 gm through 200 mesh which is 95%. For the local raw bentonite the samples are taken from purification and grinding after 100% of the sample passes through the 200 mesh. This final product of purification process is pure calcium bentonite. **Fig.60** shows the particle distribution of a sample taken from bentonite suspension prepared from this pure Ca-bentonite. From the results it is obvious that Ca-bentonite doesn't have the swelling capacity that allows it to provide good rheological properties. Particle size remains as it is after suspended in water and mixed for 20 minutes. **Fig.61** shows that when sample was treated with soda ash and aged for 24 hours it swells to some extent, and 40wt% of the particles remains as it is.

Heating/Stirring method that we introduced here allows for more swelling of the particles. In the process of heating and stirring the particles will be under thermal expansion which allows water to invade between bentonite platelets. When water be in-between the particles swell, this swelling will remain after bentonite suspension is cooled down, because the water will be trapped between the particles.

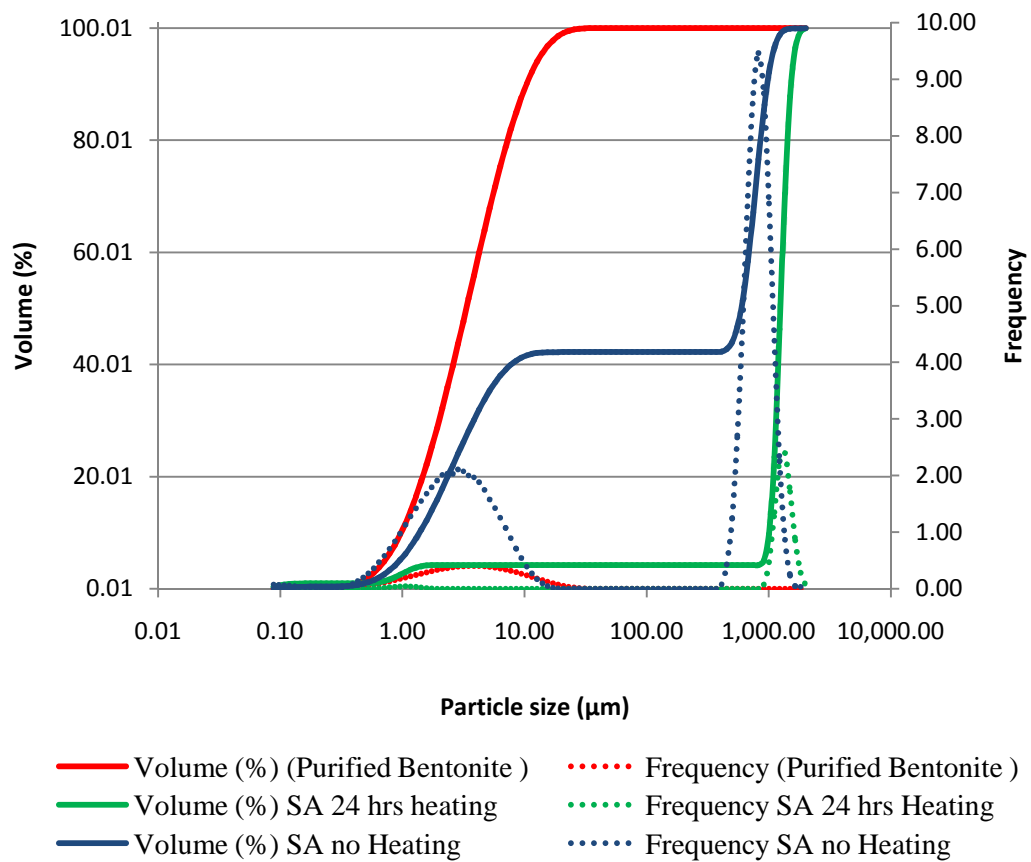
**Fig.62.** shows that sodium activation combined with heating and stirring has increased the swelling capacity significantly. The average particle size has increased from 2.4  $\sim \mu\text{m}$  for untreated bentonite to 1286.9  $\sim \mu\text{m}$ .



**Figure 60 Particle size distribution for Purified un-treated Bentonite**



**Figure 61 Particle size distribution for Purified 1 gm SA 1D aging no heating**



**Figure 62 Particle size of local bentonite with soda ash treatment**

Heating time is very critical to attain the required swelling and rheological properties. And apart from direct measuring of rheological properties this optimum time can be evaluated from particle size distribution. The maximum swelling will reflect the time required for heating/stirring treatment. A sample of pure ca-bentonite was treated with soda ash at the optimum amount of 1 gm SA to 22.5 gm of bentonite. Then the sample was subjected to heating/stirring treatment at different time intervals. The particles size distribution was measured each time. **Fig.63** combines all results and compare between them. The optimum time of heating/stirring treatments is recorded as 3 hrs this value agrees with the value evaluated by rheology measuring showed earlier in this study. **Fig.64** presents the particle size of d0.1, d0.5 and d0.9 increasing due to heating/stirring treatment, it enlarges until reaches 3 hours then it goes unchanged from 3 hours up to 24 hours. That's means the particles reach the maximum swelling at 3 hours of heating/stirring treatment.



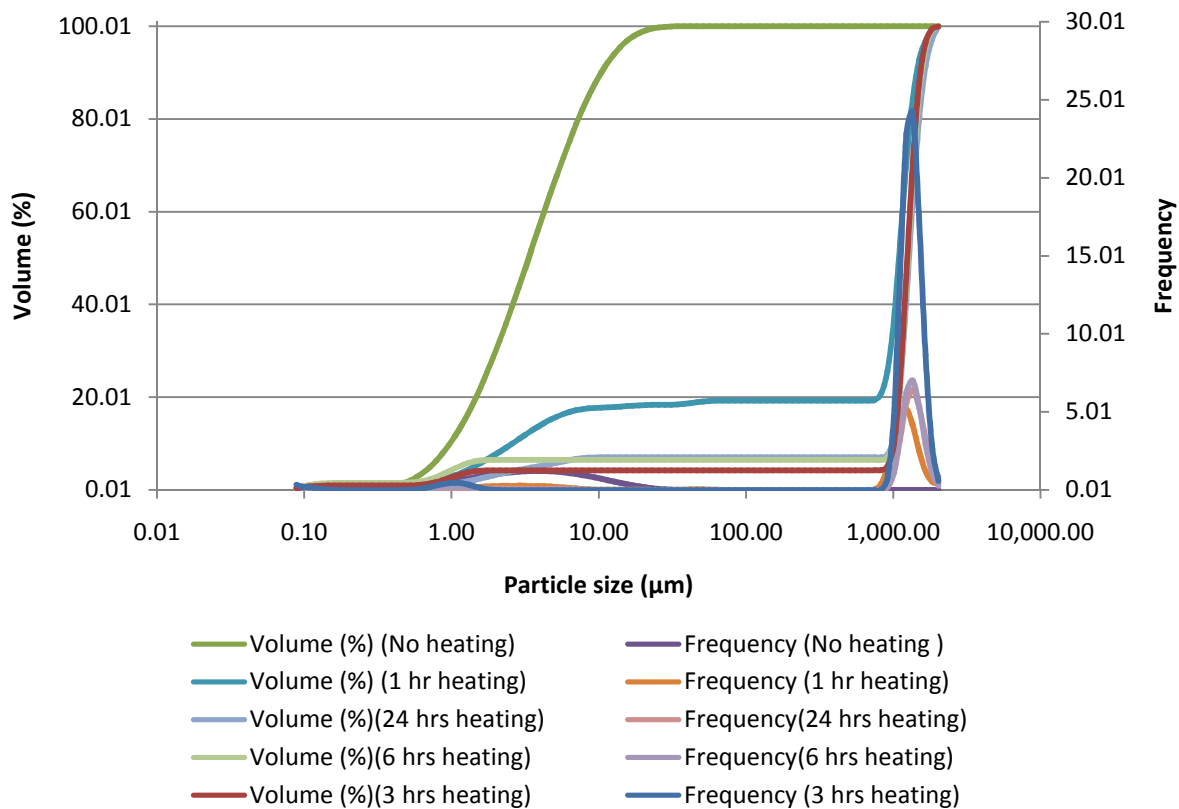


Figure 63 Particle size distribution for Purified 1 gm SA 1Daging 3hrs heating

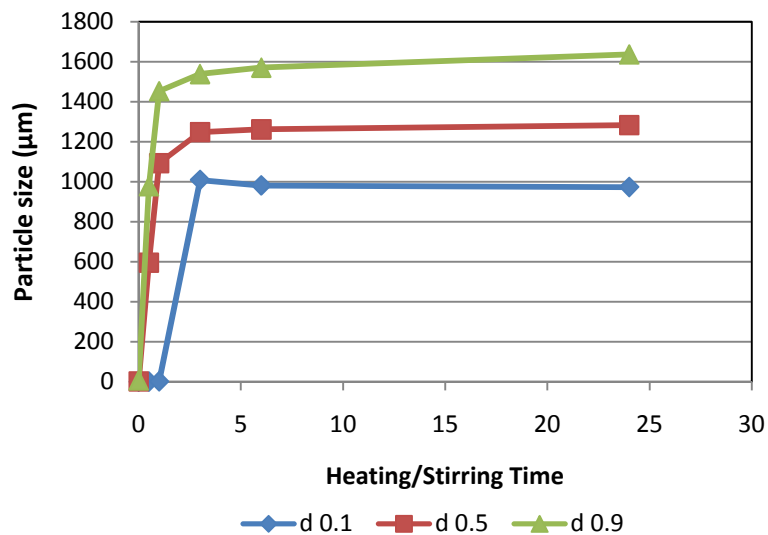


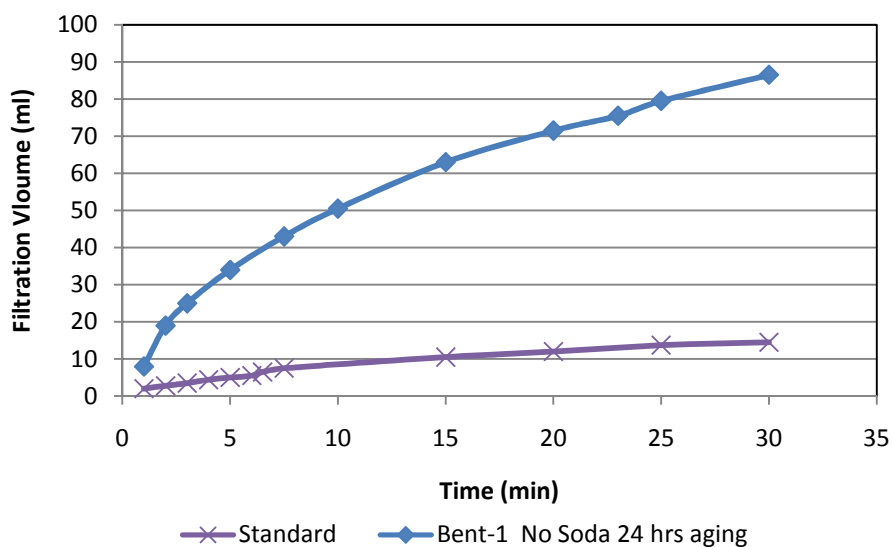
Figure 64 Particle size change due to heating time

## 5.5 Filtration

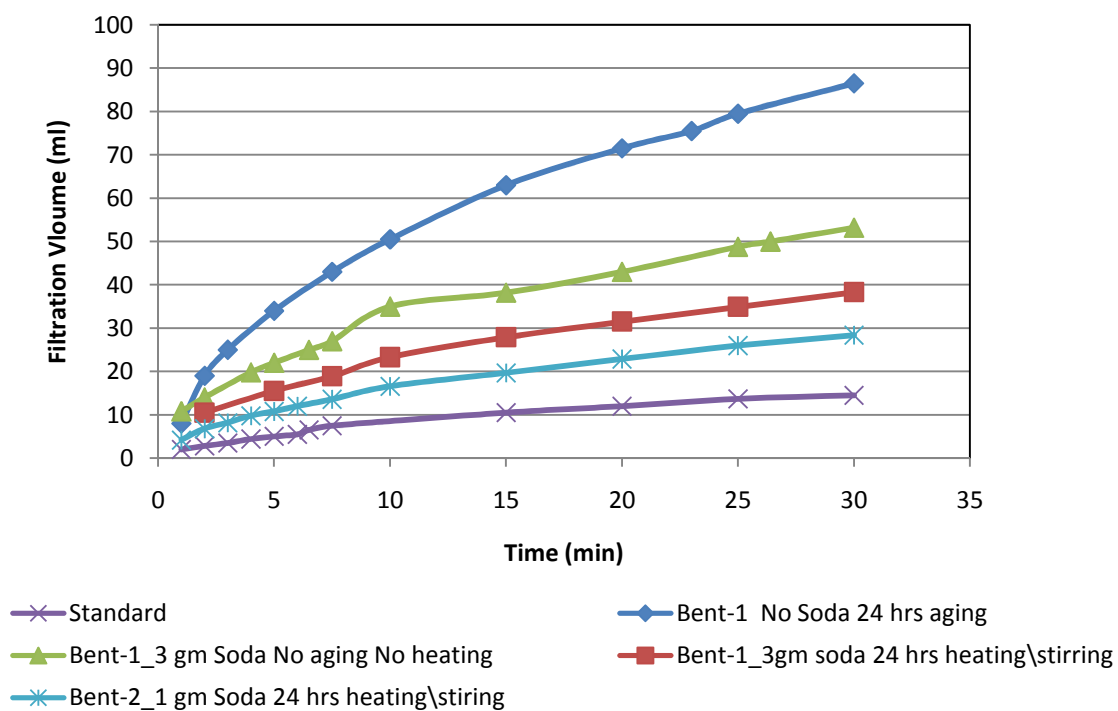
Filtration test is conducted to both raw bentonite mud and to the standard bentonite base mud. The test was aiming at evaluating the amount of water that bentonite base mud will lose to the formation. Samples prepared by mixing 22.5 gm of bentonite in 350 ml of water for 20 minutes and left to age for 24 hours. Mud sample then is put in the filter press container and subjected to 100 psi at the top. The filtrate is collected in a graduated cylinder and recorded in time intervals up to 30 minutes.

Raw untreated bentonite exhibited very high filtration; it lost 70 ml of water in 30 minutes. This value is much higher than API recommended value of maximum 15 ml within 30 minute. The standard bentonite tested gave 14.5 ml filtrate in 30 minutes. The high filtration resulted from untreated bentonite because it has very low viscosity due to the low Na/Ca ratio of the Ca-bentonite. The comparison is shown in **Fig.65**.

Activation with soda ash reduced the filtration down to 28 ml in 30 minutes. This reduction in filtration was due to the increased viscosity caused by soda ash activation. **Fig.66** shows the improvement in filtration control with addition of soda ash and heating/stirring treatment. **Fig.67** shows that Ca-bentonite after treatments formulate much better bentonite base mud. The filtration gap between the local and standard bentonite has reduced significantly. Ca-bentonite was sodium activated using soda ash at 1 gm to 22.5 gm of pure bentonite, and subjected to heating/stirring treatments for 24 hours.



**Figure 65 API Filtration for raw and standard bentonite**



**Figure 66 API Filtration for raw bentonite after soda ash treatment**

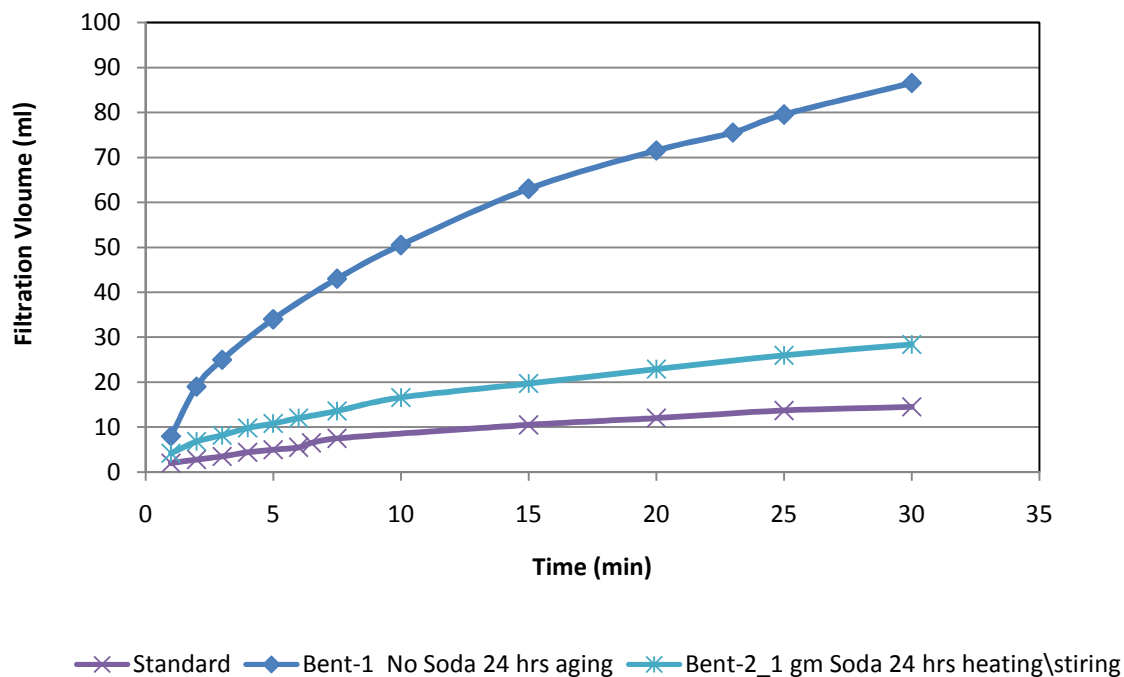


Figure 67 API Filtration for local bentonite after heating stirring treatment

## 5.6 Bentonite Rheological Properties

The rheological properties of bentonite suspension are depending on many factors, such as chemical and physical composition of the bentonite. More specifically the sodium calcium ratio, high Na\Ca ratio is inherited property of the sodium type of bentonite. The calcium type has low Na\Ca ratio, and correspondingly low swelling capacity which reflect bad suspension in water and bad rheology. Khulays bentonite, which is calcium type bentonite, when treated with soda ash combined with heating/stirring treatment, it showed great improvement in swelling and viscosity.

The rheological behavior of bentonite suspensions is known to be non-Newtonian due to the presence of fixed ionic charges on the surface of the clay particles (Gatlin, 1960; Darley and Gray, 1986; Bourgoyne et al., 1991). The viscosity of non-Newtonian fluids depends on the magnitude of the flow and the length of time the fluid has been flowing, viscosity may change with time when fluid is imposed to continuous deformation (Triantafillopoulos 1998). Shear-dependent and time-dependent rheology of fluids must be determined within the linear viscoelastic region where the rheological properties are not strain or stress dependent (Nasser and James, 2007). For full understanding of fundamental bentonite rheology more sophisticated experiments were conducted and the detailed results are shown in the following.

Bentonite suspension was prepared using two different samples; the API standard bentonite and Khulays purified bentonite. The comparison samples were in 6 wt% weight percentage of standard and local bentonite. Pure bentonite was used as non-treated and treated with soda ash using the optimum treatment amount. A comparison between treatments steps also is described in details. The standard sample was prepared according to API RP procedures by mixing 22.5 gm. of standard bentonite into 350 ml DW using high speed mixer at 11,000 rpm for 20 min, the suspension is aged for 16 hours in room temperature to allow for full hydration, then again stirred for 5 min before conducting the measurements.

Structure buildup and effect of bentonite concentrating was also studied with Na-activation using heating/stirring method. Khulays bentonite samples prepared in 3 different weight percentages 3 %, 6 % and 12 %, the samples was prepared by mixing 11.25 gm, 22.5 gm and 45 gm of local Bentonite by following the same recommended

API procedures of 20 min mixing at 11000 rpm then aged to allow hydration and pre-stir for 5 min before testing.

### **5.6.1 Linear Viscoelastic Region**

Before conducting any rheological measuring, some parameters should be selected carefully, such as the type of the test, frequency, data point intervals, time length of the test and the proper stress or strain to be applied, which should be within the Linear viscoelastic region of the fluid. Linear viscoelastic region (LVR) is defined as the region where the rheological properties are not strain or stress dependent.

The VLR can be determined in terms of elastic (storage) modulus,  $G'$ , viscous (loss) modulus,  $G''$ , and the critical stress that end the viscoelastic region. A strain or stress sweep, conducted by varying the amplitude of the input signal at constant frequency, is used to determine the limits of linear viscoelastic behavior by identifying a critical value of sweep parameter.

Two samples were taken from the standard bentonite and local bentonite to prepare a 6wt% suspension. Samples were put into concentric cylinder in DHR3 rheometer, and subjected gradually to strain percentage from 0 % up to 1000 %. It's a strain controlled oscillation, at frequency of 1.0 Hz and room temperature.

**Fig.68** and **Fig.9** shows the LVR of standard and raw local bentonite, the storage modulus and the loss modulus is plotted against the percentage of strain. Both shows gel and solid-like behavior in the linear viscoelastic region, but raw untreated bentonite has very low values of elastic modulus ( $G'$ ) less than the standard bentonite.

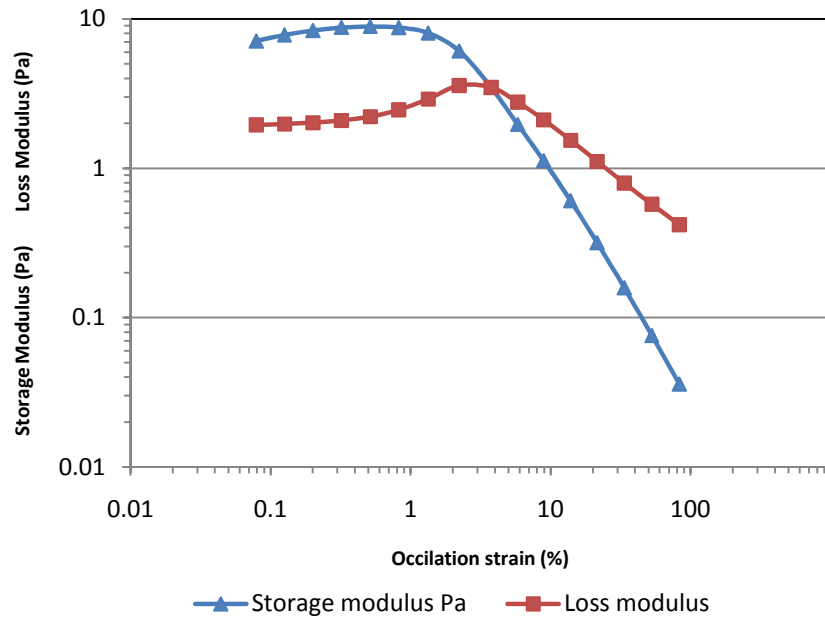


Figure 68 LVR for Standard bentonite

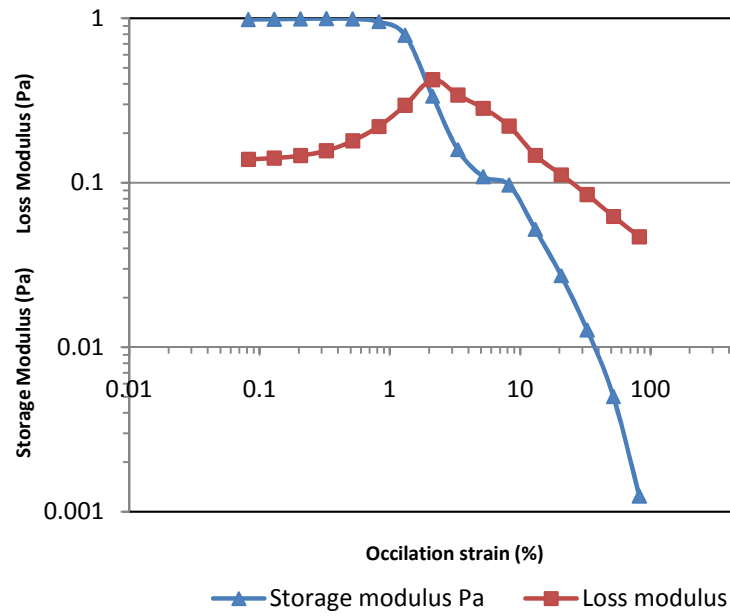
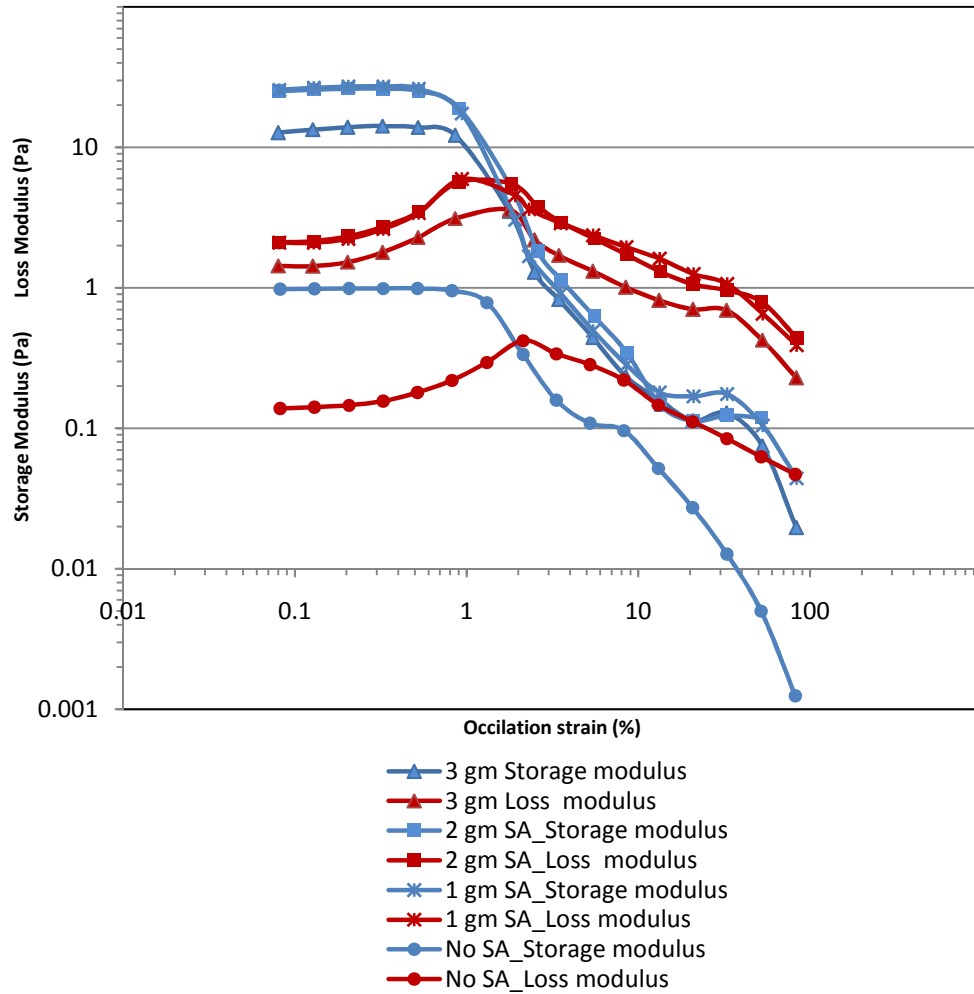


Figure 69 LVR for untreated bent-2 (without soda ash)

**Fig.70** shows the sweep frequency test carried out for activated bentonite using soda ash at different doses. The optimum value of 1 gm is proven again with this test. From **Fig.70** it is clear that 1gm of soda ash added to 6wt% of local bentonite suspension gave the highest elasticity and also it has wider linear viscoelastic region.

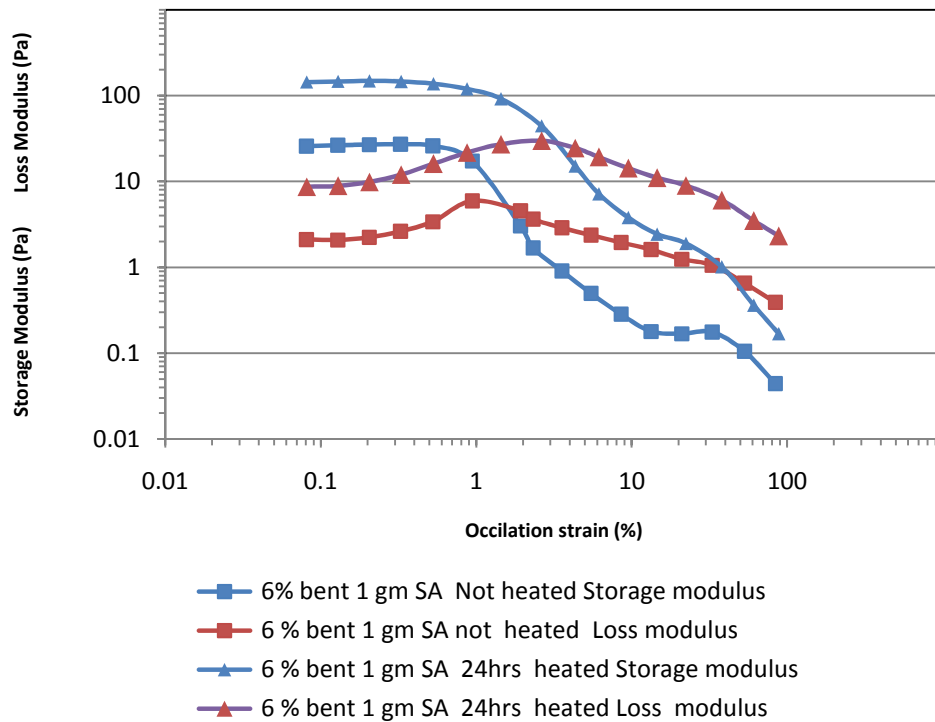


**Figure 70 Soda Ash effect on 6 % Bent-I \_ LVR**



Effect of heating/stirring treatment on elasticity of bentonite suspension is shown in **Fig.71**. Two samples, one only treated with soda ash activation at the optimum amount. And the other sample is treated with heating/stirring for 34 hours. The plot of storage modulus  $G'$  and loss modulus  $G''$  has shifted to the right side towards higher strain percentage, and also upwards to the higher elasticity. In both plots is shown, that  $G > G''$  which means that the dispersions are solid-like and have high structural strength.

Both moduli exhibit a constant value and the storage modulus continue to be higher than loss modulus ( $G' > G''$ ), after shear strain exceed the critical value and linear viscoelastic region reach to its end, the storage modulus decreases with increasing shear strain while loss modulus  $G''$  increases with strain and form a peak then decrease again, this effect is due to a strain-hardening effect, which indicate that fluid elasticity is decreased.



**Figure 71 6 % bent 1 gm SA before and after heating/stirring \_ LVR**

**Fig.72** shows the variation of linear viscoelastic region with concentration of bentonite, a fixed frequency of 1 Hz was maintained constant while the oscillation strain was automatically increased stepwise corresponding to the difference in shear stress. The storage modulus and loss modulus were measured at different strain percentage. The behavior is normal that with higher concentration the linear viscoelastic region decreases. The effect of heating\stirring is obvious in **Fig.73**, LVR becomes wider after treatment. **Fig.74** shows heating\stirring for 12 hours has the same effect of 24 hours and storage modulus is higher above the untreated raw bentonite. All the tests were conducted within the viscoelastic region to be sure, the samples are safely within the linear viscoelastic region.

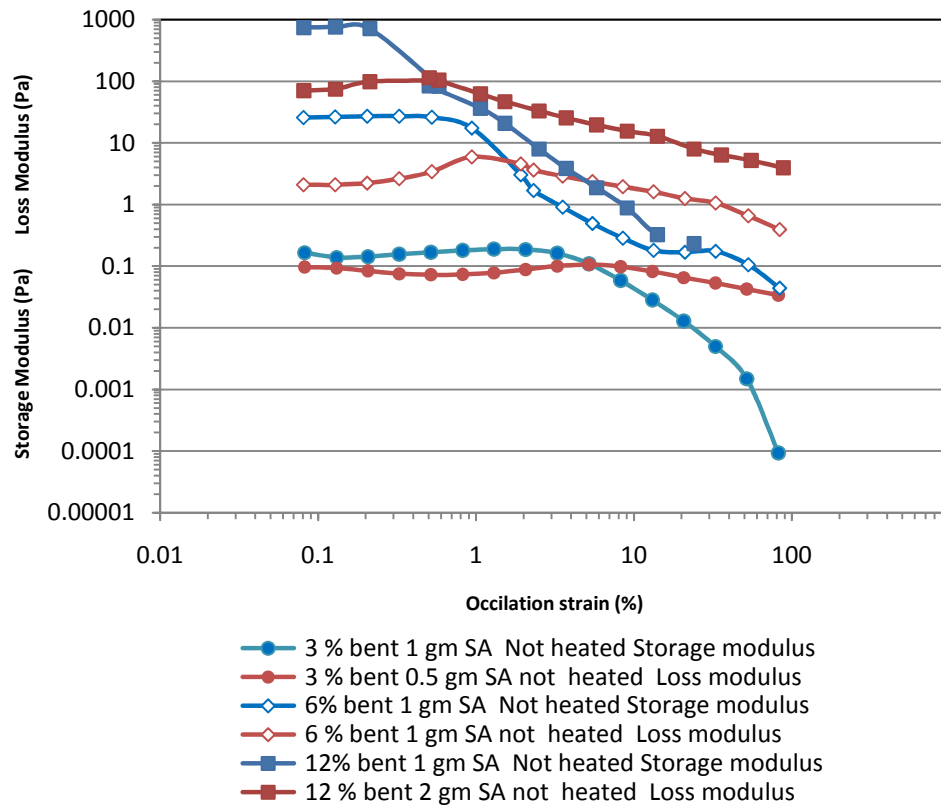


Figure 72 bent different percentage and SA before and after heating\stirring treatment \_ LVR

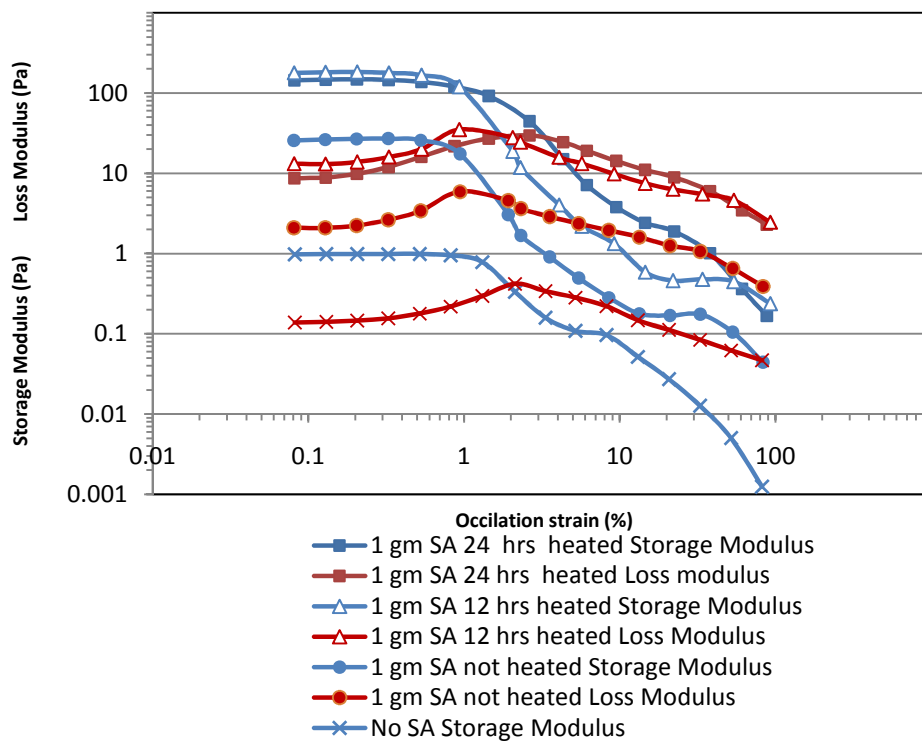


Figure 73 effect of heating/stirring on LVR

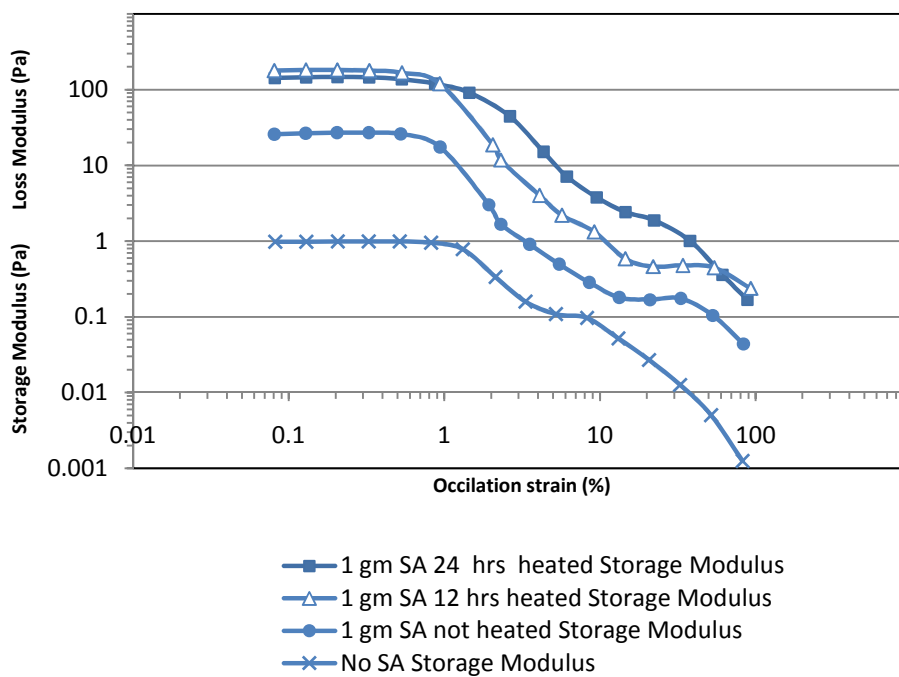


Figure 74 Effect of heating/stirring on elasticity (storage modulus)

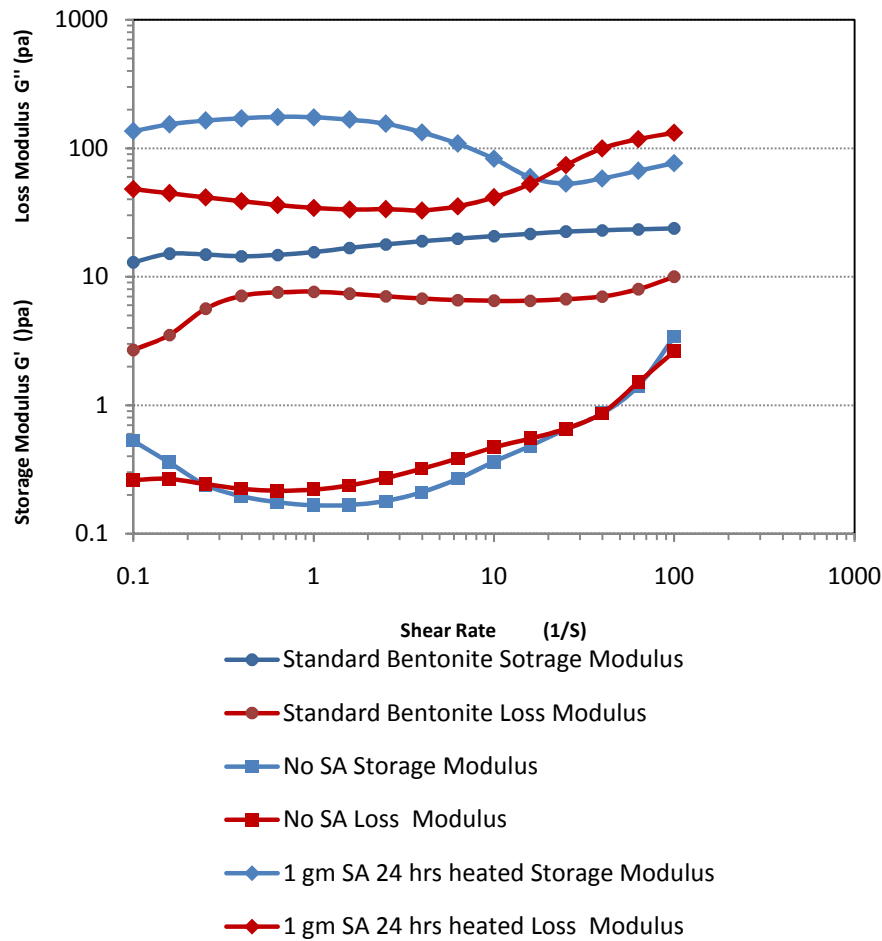
### 5.6.2 Dynamic Test (Frequency Sweep Oscillation)

The structure of bentonite suspension can be characterized using a frequency sweep at a strain below the critical strain defined from the strain sweep, to make sure that the rheological behavior of the samples during the test is independent of imposed stress or strain levels. The frequency sweep test was carried out for Khulays bentonite as raw, untreated, and treated at all the different treatment stages. Measurement was performed over a range of oscillation frequencies (0.1 to 100 rad/s) at constant oscillation amplitude at room temperature.

**Fig.75** shows the results of frequency sweep in a dynamic test. The frequency was maintained constant at 1 Hz, strain was maintained at 10% to be within the LVR and make sure that the rheological behavior of the samples during the test is independent of imposed stress or strain levels. The stress is changed automatically, angular frequency from 0.1 rad/s to 100 rad/sec, at room temperature. The standard bentonite (the two curves at the middle of the plot) shows that standard bentonite suspension of 6wt% has storage modulus ( $G'$ ) higher than the loss modulus ( $G''$ ) 5 to 1 through all the plot, that's means it has a gel or solid-like behavior independent of shear rate. Raw untreated bentonite has high loss modulus at the most of the plot, this indicates it behaves like liquid at all shear rates and it shows very little values which indicate very low viscosity nearly water. Treated local bentonite has improved substantially and storage modulus increased from less than 1 Pa to more than 150 Pa after local bentonite was activated using soda ash and subjected to heating/stirring treatments. Also loss modulus increased from less than 1 Pa to 50 Pa, and elasticity remain higher represented in higher storage modulus compared to loss modules by 3 to 1. This improvement shows the effect of

treating and activating local bentonite, the plot shows an improvement of 100 times compared to untreated bentonite, and changed the local bentonite suspension from liquid-like behavior to solid-like behavior.

**Fig.75**, Fig.76and **Fig.77** shows degree of soda ash activating with different does. A comparison between 0.5 gm, 1gm, 2gm and 3 gm of soda ash, with and without heating/stirring treatment. Again 1 gm soda ash to 22.5 gm bentonite has the best results and heating/stirring treatment enhanced the suspension rheology.



**Figure 75** Frequency sweep test for treated and untreated bentonite

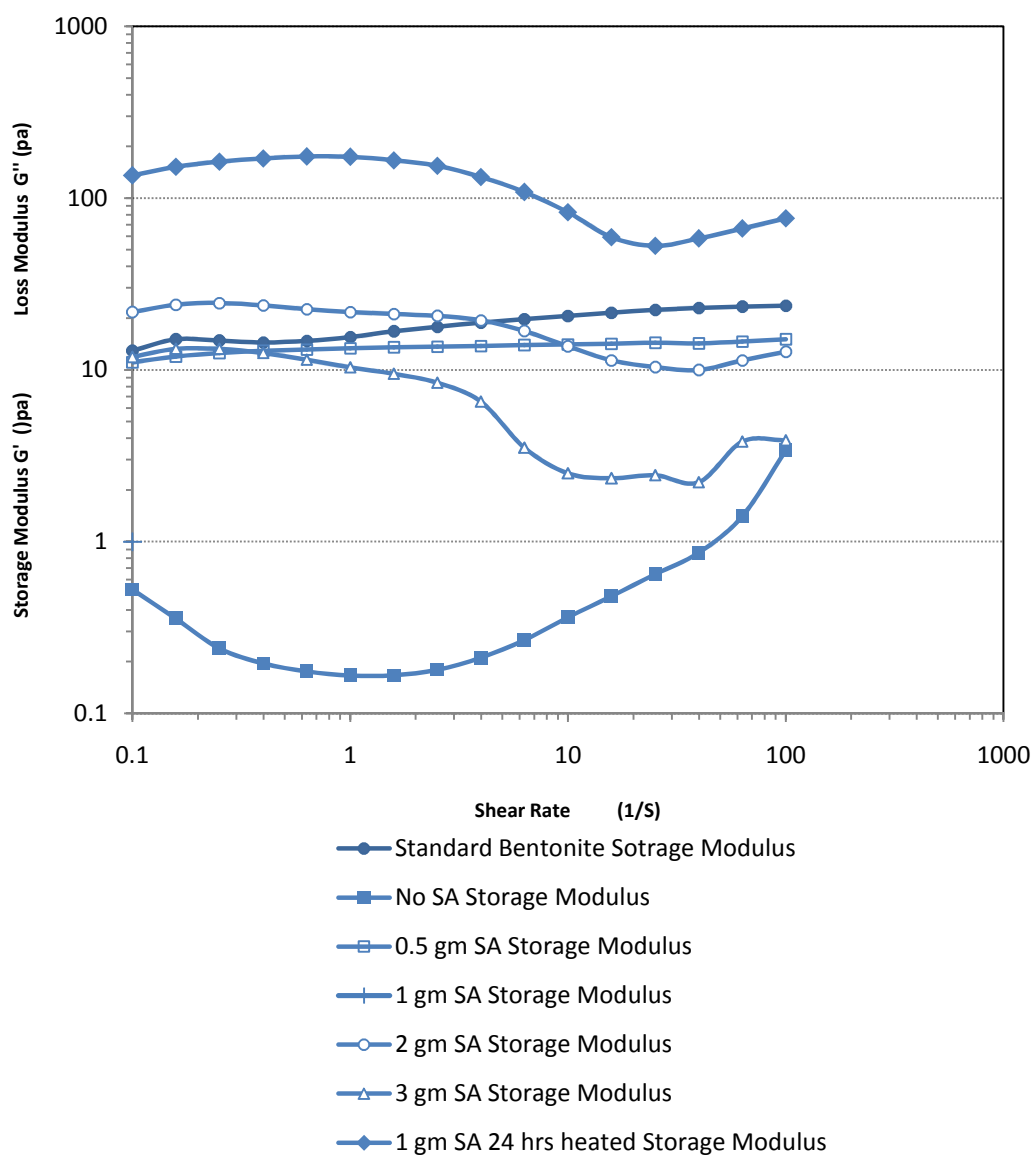


Figure 76 Frequency sweep test, dependant of  $G'$  on the amount of soda ash and heating treatment

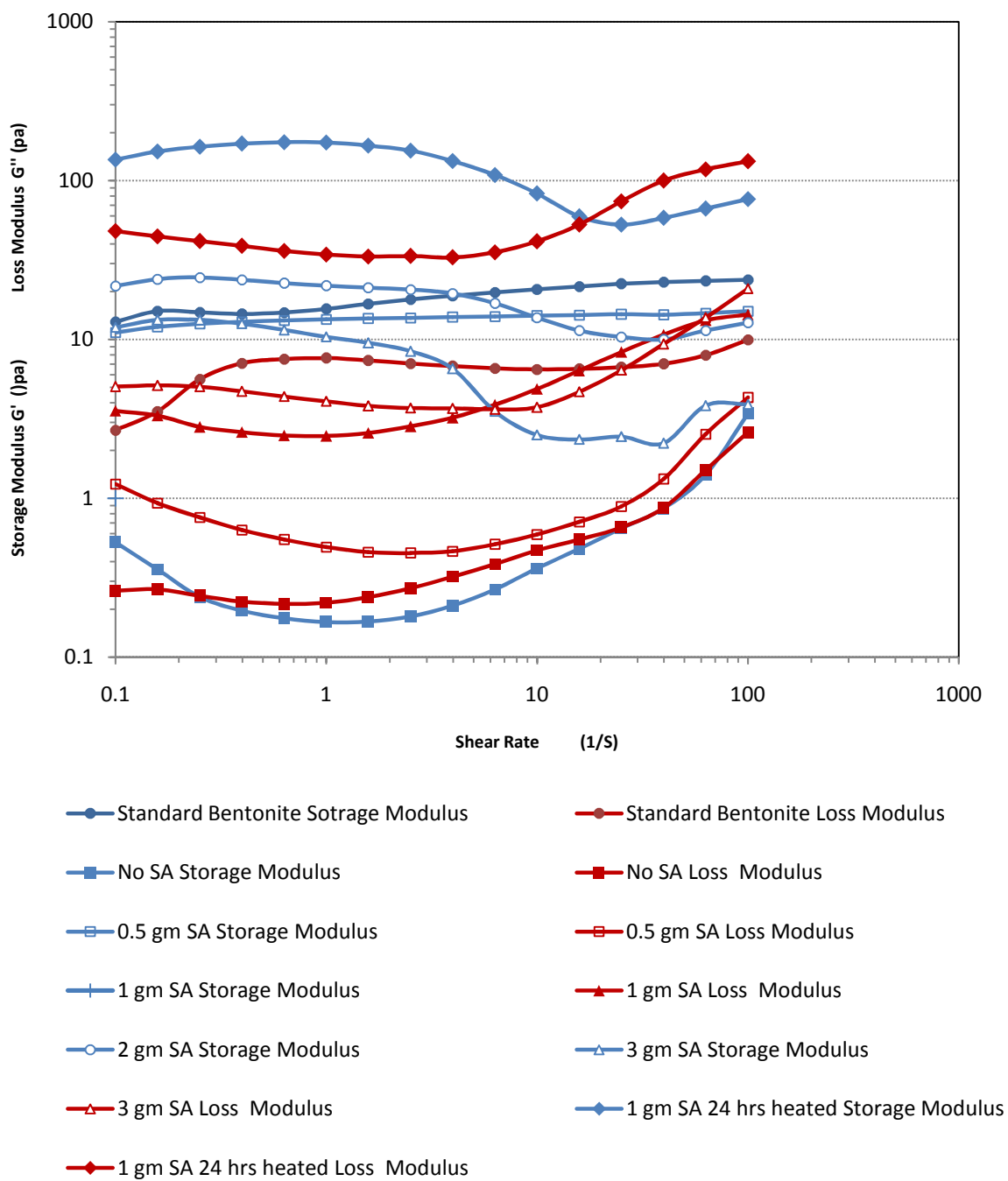
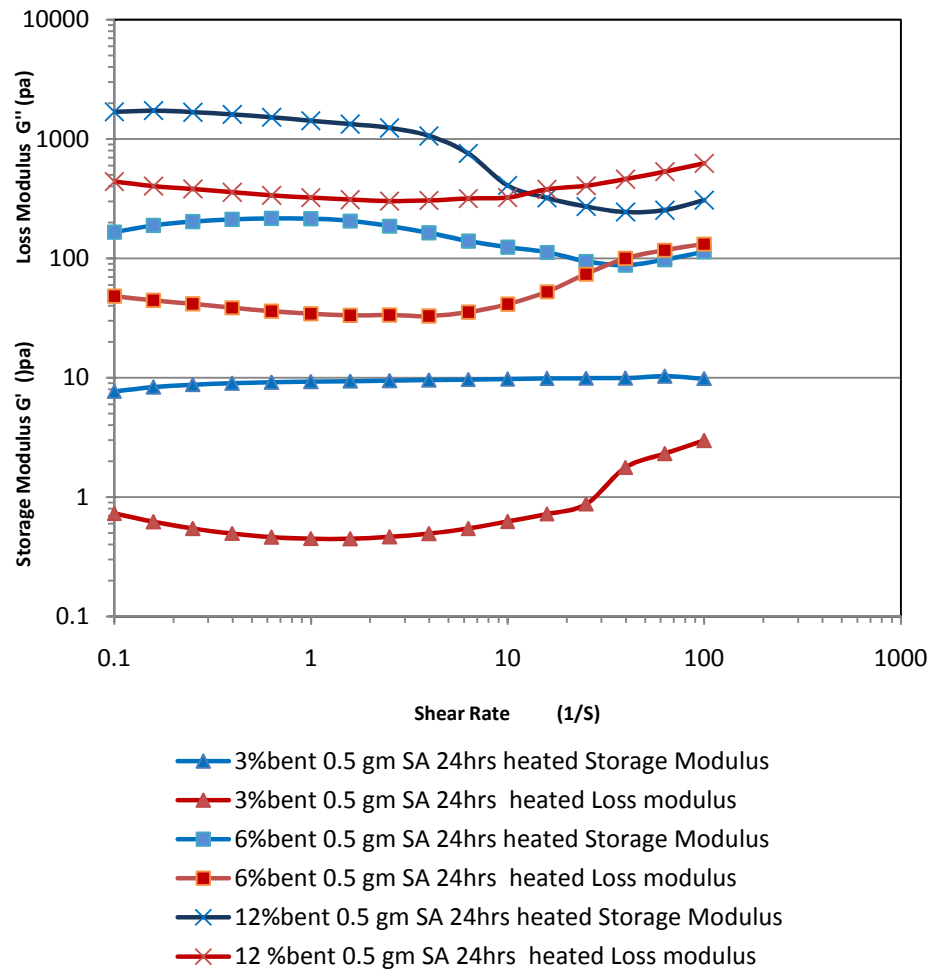


Figure 77 Frequency sweep test comparison for the amount of soda ash

Since bentonite concentration also affects the behavior of the suspension, diluted and heavier systems were prepared from the treated bentonite. Dynamic shear test was conducted in concentric cylinder; samples were subjected to angular frequency from 0.1 rad/s to 100 rad/sec, at room temperature. Under maintained frequency at 1 Hz, strain was maintained at 10% to be within the LVR. The heavier the suspension is the narrower the range between elasticity and viscous modules. Results on **Fig.78** shows that all systems has solid-like behavior, but heavier system tends to change to liquid-like behavior at very high shear rates above 15 1/s for 6wt% and 100 1/s for 12wt%.



**Figure 78 Frequency sweep test, dependant of bentonite concentration**



Heating/stirring treatments effect on the structure of bentonite suspension is shown in **Fig.79**. Great improvement from 1 up to 100 Pa is shown on the plot, even higher than the standard bentonite which gave around 15 Pa. heating time from 12 hrs and 24 hours are shown, both achieved the same results. The effect of heating/stirring treatment is more clearly shown in **Fig.80** both elasticity and viscous modulus are shown, they are shifted upwards in the plot towards higher values, while the storage (elasticity) modulus become larger, this means that fluid is becoming more solid-like behavior and elasticity is increasing with the treatment. It shows also that 12 hours and 24 hours had the same effect and results in the same gel structure, the plots of these two samples are identical.

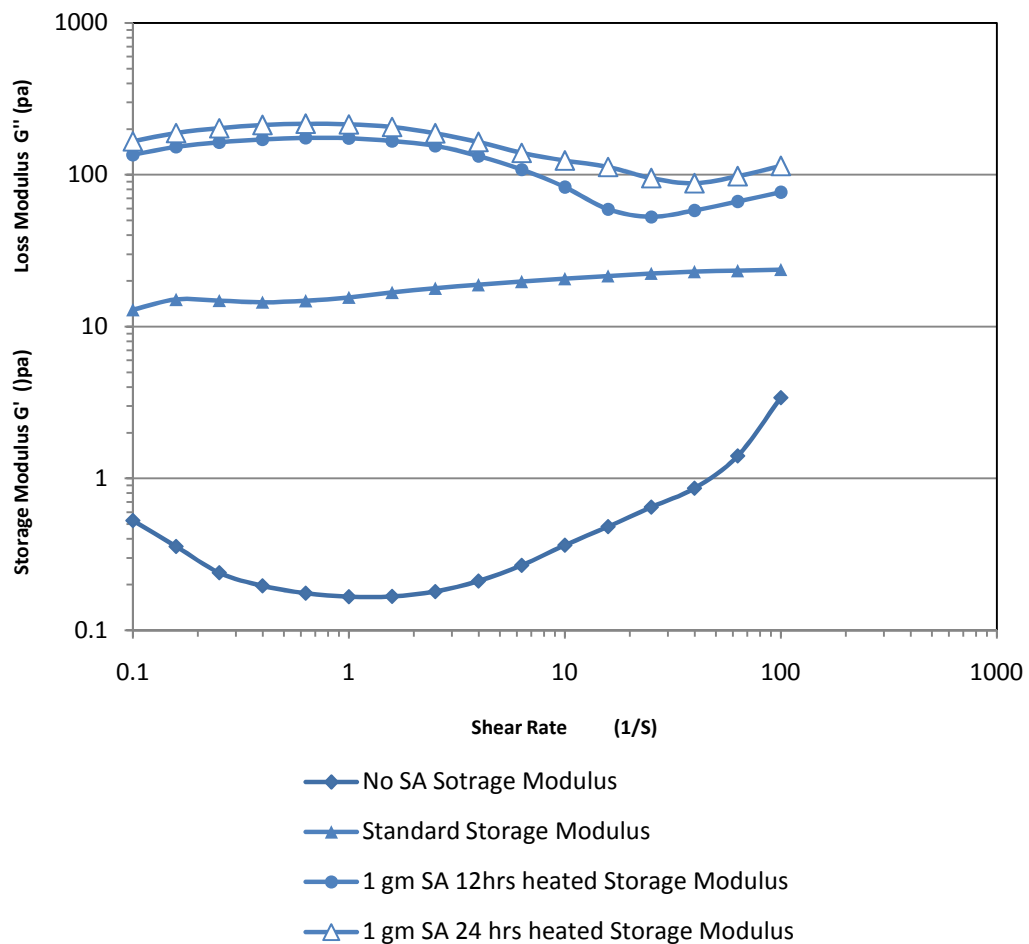


Figure 79 Frequency sweep test, dependant of  $G'$  on heating time

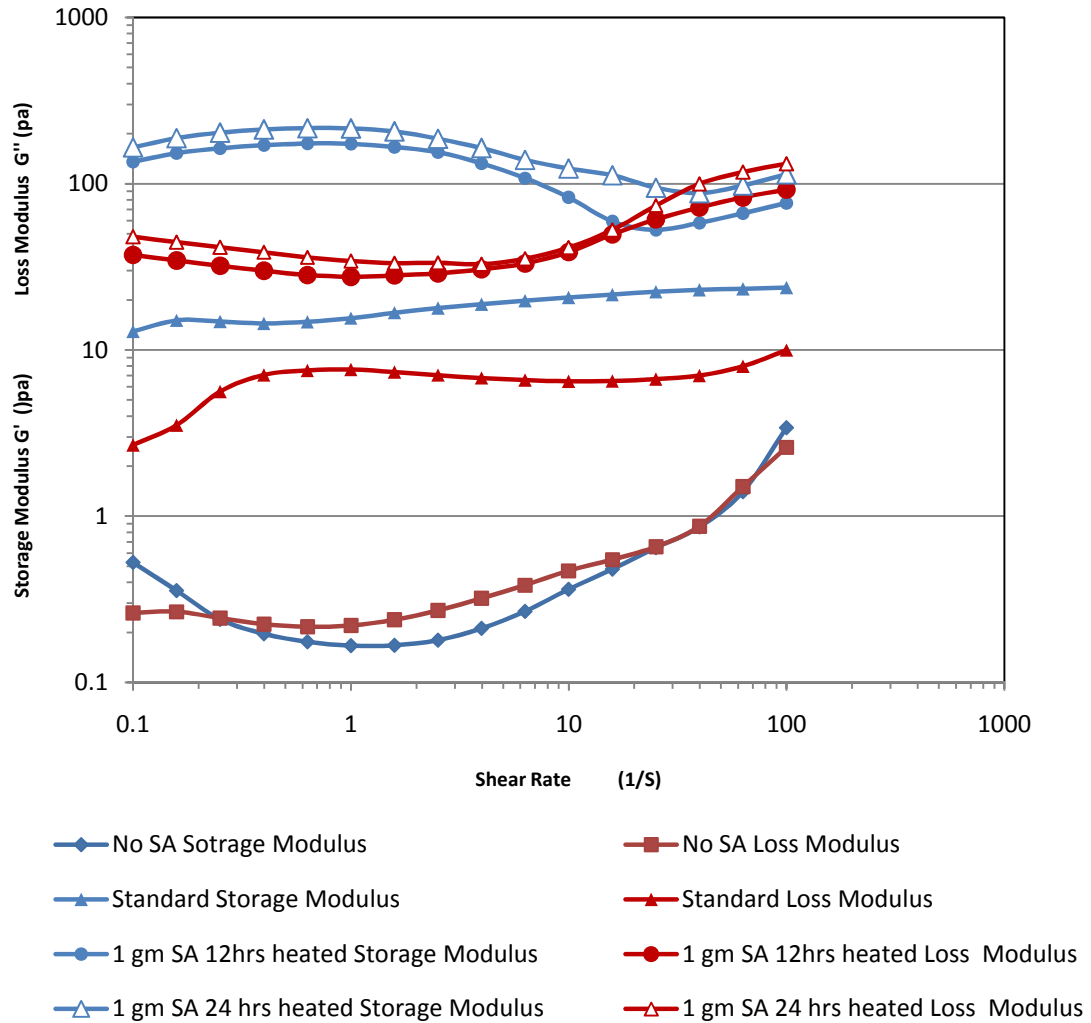


Figure 80 Frequency sweep test comparison for standard, treated and untreated bentonite

### 5.6.3 Flow Sweep Test

Bentonite suspension in water forms a non-Newtonian viscous fluid. Apart from the concentration, temperature and the other factor, the viscosity of the fluid is depending on the rate of shear that it was subjected to. In drilling fluid bentonite based mud is stirred at high speed to formulate the mud and pumped into the well. Inside the well mud is will be subjected to a different circulation speeds and pressure, beside the shear applied by

rotation of the drilling string. A flow sweep test is conducted to bentonite suspension at 6wt% before and after being activated and treated with heating/stirring process. Also diluted and heavy sample were tested to give viscosity profile of the different systems through low shear rates and high shear rates.

At low shear rate the flow sweep gives information on the viscosity behavior that occurred during certain condition such as sedimentation and separation during storage and structure retention.

**Fig.81** shows the results of flow sweep conducted for purified bentonite after being activated with soda ash at 0.5 gm, 1 gm, and 3 gm, to be compared with the standard bentonite sample prepared at the same concentration 6wt% with the same procedures. Untreated bentonite has very low viscosity profile decaying from less than 2 Pa.s. Sodium activated samples shows some improvement from 0.5 gm to 1 gm soda ash, then with 3 gm the affect was reversed. The amount of 1 gm soda ash result in viscosity profile almost tracking the standard bentonite viscosity profile through the medium shear rates, and slightly decrease below the standard at the low and high shear rates.

**Fig.82** combine the results with the heated/stirred samples, and shows that when samples were subjected heating/stirring treatment it resulted in much higher viscosity profile, even way above the standard bentonite. Again heating/stirring for to 12 hours and 24 hours gave identical viscosity profiles

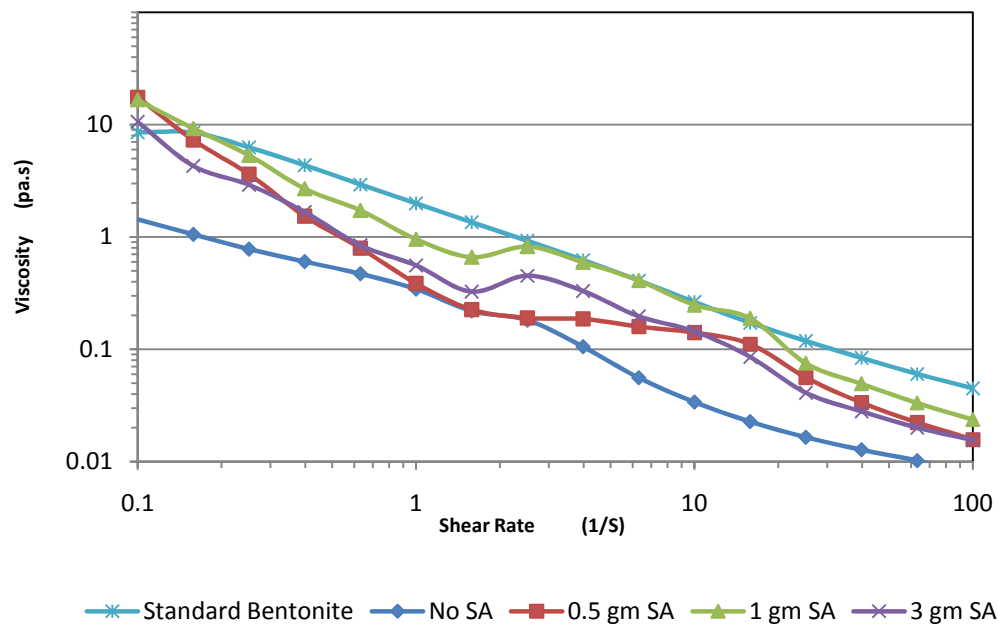


Figure 81 Flow sweep test of soda ash activated bentonite

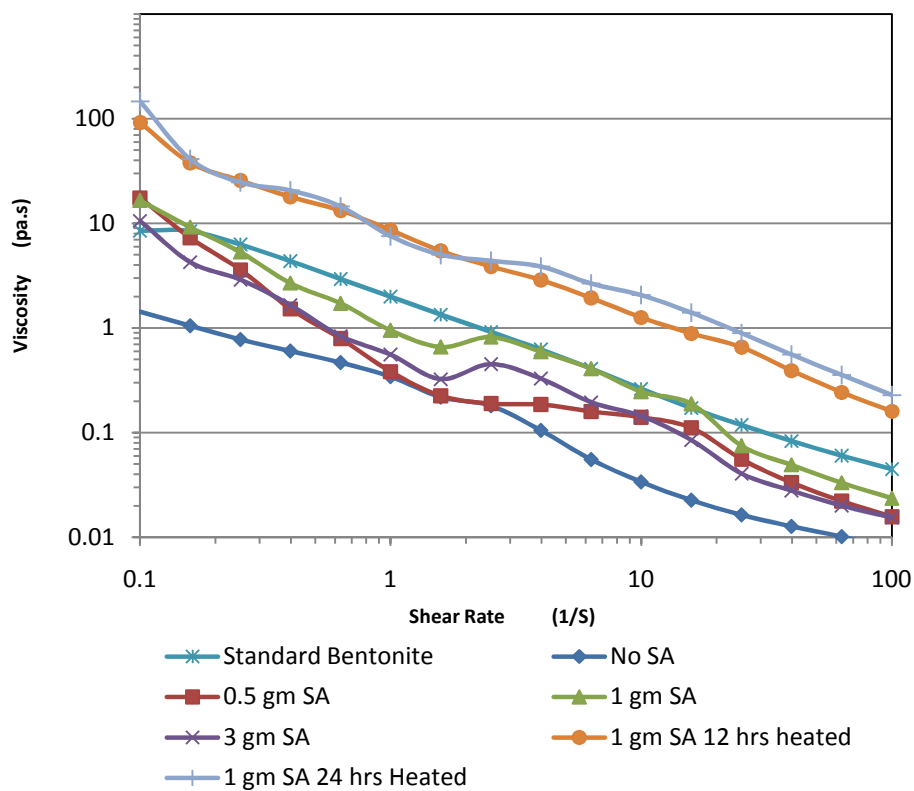
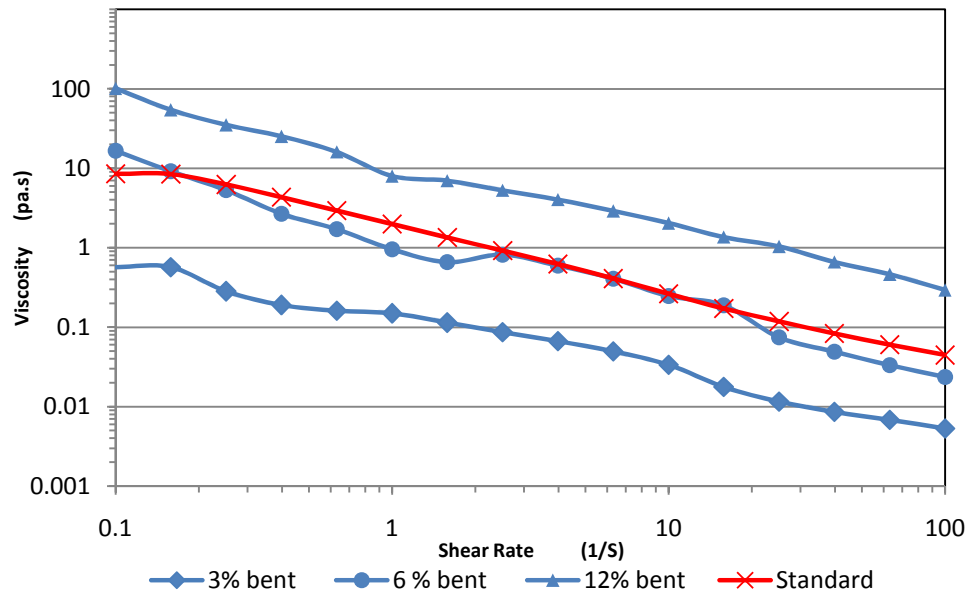


Figure 82 Flow sweep test comparison for soda ash amount and heating time

**Fig.83** shows viscosity profile changing due to increasing and decreasing of bentonite concentration when samples were treated with soda ash and Fig.84 with heating/stirring treatment. The results showed that 6%wt of bentonite doesn't fully saturate the water suspension, and the fluid can take more bentonite. Adding more bentonite higher than 6wt% of treated samples does not cause any precipitation or settling. This was indicated by the increasing of viscosity profile at higher concentration.

Heating/stirring treatment after soda ash activation can raise the viscosity profile for local bentonite to a level that in some cases we can use half of the bentonite amount to achieve the same properties. **Fig.85** shows that only 3wt% of treated bentonite can be form a viscous fluid with viscosity profile higher than the 6wt% standard bentonite through all shear rates.



**Figure 83** Flow sweep test for standard bentonite and activated bentonite at different wt concentration (not heated)

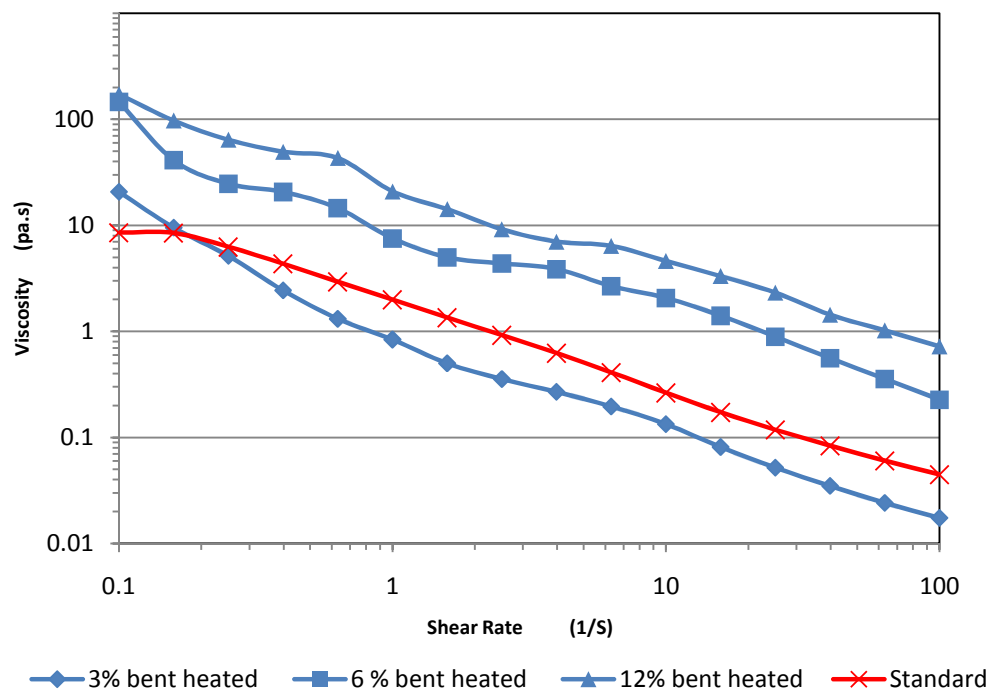


Figure 84 Flow sweep test for standard bentonite and activated bentonite at different wt concentration (24 hrs heated)

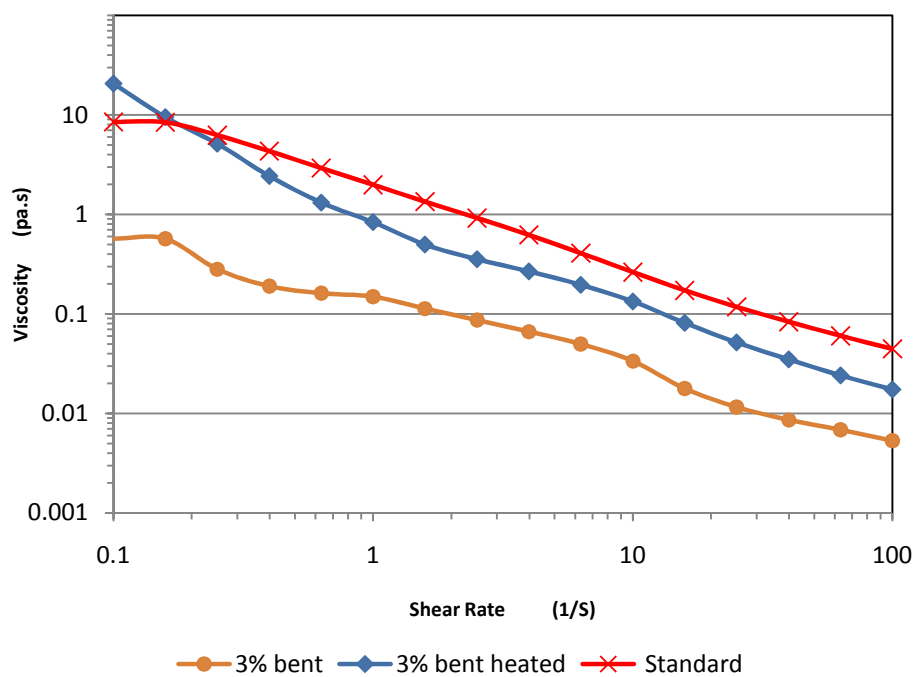


Figure 85 Flow sweep test for standard bentonite and activated 3 wt % bentonite before and after 24 hrs heating

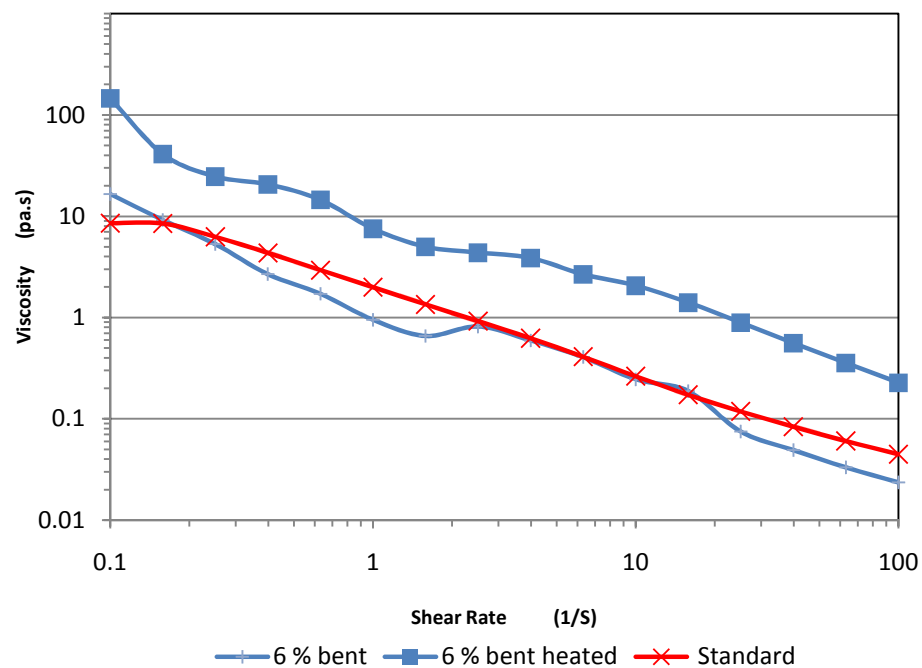


Figure 86 Flow sweep test for standard bentonite and activated 6 wt % bentonite before and after 24 hrs heating

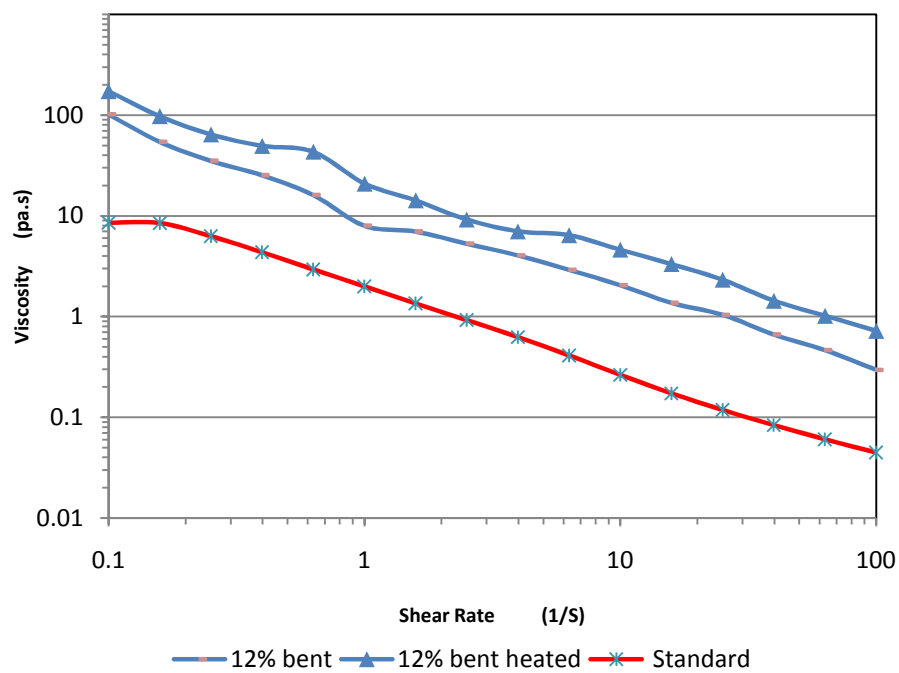
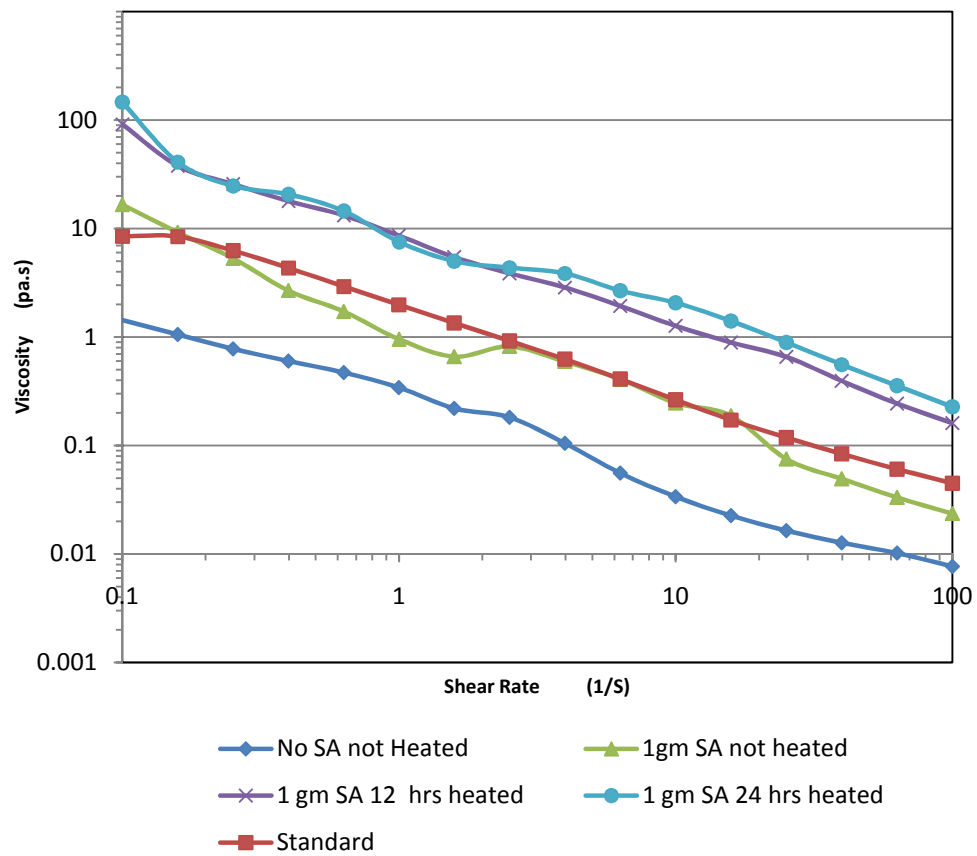


Figure 87 Flow sweep test for standard bentonite and activated 12 wt % bentonite before and after 24 hrs heating

**Fig.88** shows combined results of flow sweep test to untreated, soda ash treated and heating/stirring treated bentonite. Viscosity profile is respectively improving with the treatment stages.



**Figure 88** Flow sweep test, dependant of viscosity on heating time



#### 5.6.4 Time Sweep

Dynamic time sweep or time ramp test was conducted to define the Time dependent Thixotropy, shear thinning and the stability characteristics of the of the bentonite suspension. The viscosity is monitored at constant frequency amplitude and constant temperature for a period of time until it reaches flattened profile.

**Fig.89** shows result of oscillatory time sweep tests conducted to samples of bentonite 6wt% treated with soda ash at 0.5gm, 1gm,2gm and 3 gm. The data gathered through the time ramp gave the change with time under fixed strain rate at room temperature subjected for 10 minutes. Untreated bentonite gave very low profile from the beginning of the time, activated samples showed increasing in viscosity from 0.4 up to 0.8 Pa.s. The optimum amount of soda ash that does not overdose the bentonite treatment was proven to be 1 gm soda ash added to 22.5 gm of soda ash at 6wt% solution. Based on **Fig.89** the viscosity exhibit an increasing at the very beginning of the time then it decay in a uniform profile. The profile increased with soda ash addition up to 1 gm, then with 2gm and 3 gm it decreasing and giving lower viscosity profiles. That's due to the excessive amount of soda ash.

**Fig.90** shows the result of time ramp test for heating/stirring treatment, at different stages from 1 hour to 24 hours. Viscosity profile starts decaying from higher values and flattened at much higher viscosity after 10 minutes, it increased from 0.1 Pa.s to 1.6 Pa.s through the heating/stirring treatment.

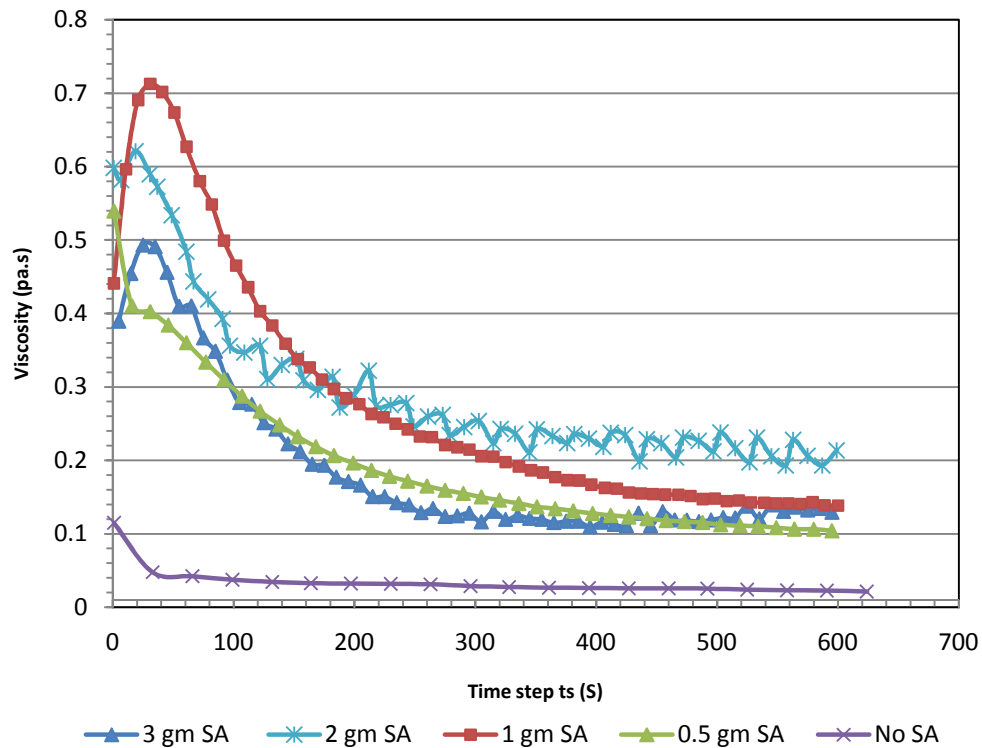


Figure 89 Time Sweep Soda Ash effect on 6 % Bent-2

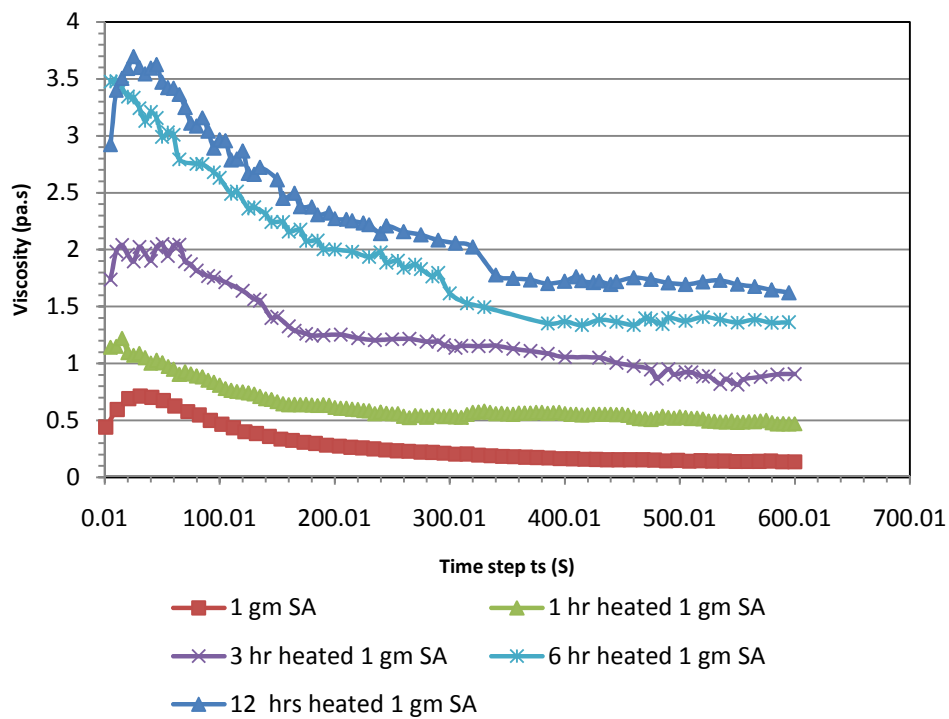


Figure 90 Time sweep, time effect on 6wt% bent-2 after soda ash and heating

## 5.7 Drilling Fluid Formulation

Khulays bentonite purified using the proposed procedures of sedimentation and wet sieving. The obtained pure bentonite upgraded through the thermal treatment, and the final product was used in a selected drilling fluid formulation. Two types of formulation were prepared, 80% and 5% bentonite base mud. The 80% bentonite base mud is used for the upper section of the well before reaching the pay zone. This type is called the drilling fluid, in which the formation damage caused by filtration is not a critical as the pay zone. The second formulation is called drill-in fluid, it is using very less bentonite and used to drill the oil formation (pay zone) where permeability is highly concerned and damages caused by filtration is not allowed. Each formulation was prepared using both standard bentonite and local treated bentonite for comparison.

### 5.7.1 Bentonite Base Mud

The first formulation used bentonite, caustic soda, xanthan gum and calcium carbonate. The weigh percentages are shown in **Table.20**. Components were added in the mentioned sequence and with stirring for 5 min, 20 min, 10 min, 5 min and 5 min correspondingly. The mud was aged for 24 hours to allow for complete hydration and to give the best results. A Fann35 speed dial viscometer was used to measure the viscosity of the mud and detailed reading are shown in **Table.22**. The density of the mud was measured using mud balance. API filtration was collected through 30 minutes, and mud cake thickness was reported.

**Table 20 Formula 1-A (Standard Bentonite)**

Formula 1-A Component	Lab unit		Field unit
Water	350	ml	bbl
Soda Ash	1	gm	ppb
Bentonite (std)	20	gm	ppb
Caustic Soda	0.75	gm	ppb
Xanthan Gum	0.75	gm	ppb
CaCo3	1	gm	ppb

**Table 21 Formula 1-B (bent-2)**

Formula 1-B Component	Lab unit		Field unit
Water	350	ml	bbl
Soda Ash	1	gm	ppb
bent-2	20	gm	ppb
Caustic Soda	0.75	gm	ppb
Xanthan Gum	0.75	gm	ppb
CaCo3	1	gm	ppb

**Table 22 Viscosity and API filtration test for Formula 1A & 1B**

Property	Standard bent	bent-2
----------	------------------	--------

600	65	60
300	45	50
200	36	45
100	25	35
6	10	30
3	8	38
10 sec	8	49
10 min	40	55

Mud weight	10.9	10.9
PH	12	12

Av	32.5	30
PV	20	10
YP	25	40
Filtrate Volume (ml)	9	30
Mud cake thickness	3	4

The second formulation was prepared using bentonite, soda ash, caustic soda, KCl, xanthan gum, calcium carbonate and starch. The components shown in **Table.23** were added in the mentioned sequence and with stirring time for 20 min, 5 min, 5 min, 10 min, 10 min, 5 min and 5 min correspondingly. A Fann35 speed dial viscometer was used to measure the viscosity of the mud and detailed reading are shown in **Table.25**. The density of the mud was measured using mud balance. API filtration was collected through 30 minutes, and mud cake thickness was reported.

**Table 23 Formula 2-A (Standard Bentonite)**

Formula 2-A Component	Lab unit		Field unit
Water	350	ml	bbbl
Soda Ash	0.5	gm	ppb
Bentonite (std)	6	gm	ppb
Soda Ash	0.5	gm	ppb
Caustic Soda	1	gm	ppb
KCl	18	gm	ppb
Xanthan Gum	1	gm	ppb
CaCo <sub>3</sub>	5	gm	ppb
Starch	6	gm	ppb

**Table 24 Formula 2-B (bent-2)**

Formula 2-B Component	Lab unit		Field unit
Water	350	ml	bbl
Soda Ash	0.5	gm	ppb
Bent-2	6	gm	ppb
Soda Ash	0.5	gm	ppb
Caustic Soda	1	gm	ppb
KCl	18	gm	ppb
Xanthan Gum	1	gm	ppb
CaCo <sub>3</sub>	5	gm	ppb
Starch	6	gm	ppb

**Table 25 Viscosity and API filtration test for Formula 2A & 2B**

Property	Standard bent	bent-2
----------	------------------	--------

600	23	22
300	15	15
200	12	11
100	8	8
6	3	3
3	2	2
10 sec	3	3
10 min	12	34

Mud weight	8.5	8.5
PH	12.6	12.6

Av	11.5	11
PV	8	7



### 5.7.2 HP/HT Test for Barite formula

In mud formulating barite is used as a weighting material and it is used only in the upper section of the drilling before reaching the oil pay zone. Barite if used to drill the oil zone it will lead to serious formation damage. The treated local bentonite was used to prepare a drilling fluid with barite as a weighting material. The local formula compared to a drilling fluid with the same formula that uses the standard bentonite. The detailed formula is shown in **Table.26**. The formulation was containing caustic soda to increase the pH of the mud, and Carboxymethyl cellulose (CMC) to increase the viscosity of the mud, in addition to barite which is used as a weighting material. The mud formulations were tested at two different elevated temperatures, 160 °F and 200 °F. The detailed readings are shown in **Table.27** and **29**.

**Table 26 Barite formula with standard bentonite**

Component	Lab unit	
Water	350	ml
Standard Bentonite	20	gm
Soda Ash	1	gm
Caustic Soda	0.5	gm
High Visc CMC	1.5	gm
Low Visc CMC	3.5	gm
Barite	160	gm

**Table 27 Viscosity and API filtration test for barite formula with standard bentonite**

Property	160 oF	200 oF
----------	--------	--------

600	123	127
300	82	86
200	66	68
100	45	47
6	15	13
3	14	13
10 sec	15	15
10 min	48	44

Mud weight	11.1	11.1
PH	11.6	11.2

Av	61.5	63.5
PV	41	41
YP	41	45

**Table 28 Barite formula with Local treated bentonite**

Component	Lab unit		Field unit
Water	350	ml	bbl
Local Bentonite	20	gm	ppb
Soda Ash	1	gm	ppb
Caustic Soda	0.5	gm	ppb
High Visc CMC	1.5	gm	ppb
Low Visc CMC	3.5	gm	ppb
Barite	160	gm	ppb

**Table 29 Viscosity and API filtration test for barite formula with standard bentonite**

Property	160 oF	200 oF
----------	--------	--------

600	127	136
300	85	94
200	68	76
100	48	54
6	23	35
3	22	30
10 sec	29	38
10 min	99	140

Mud weight	11.1	11.1
PH	11.6	11.2

Av	63.5	68
PV	42	42
YP	43	52

## CHAPTER 6

### CONCLUSIONS AND RECOMMENDATIONS

Khulays Local bentonite was investigated for possible use in drilling fluid formulations. The local bentonite was characterized to evaluate its chemical composition and physical properties. Different purification methods were used to select the best way of extracting a pure bentonite and remove the non-clay impurities. The soda ash treatment was used to increase Na/Ca ratio of the local calcium bentonite to attain an improved swelling and rheological properties. A novel method of combined heating and stirring was introduced. Using this new method increased the swelling capacity significantly. The rheological properties enhanced and achieved the target of standard API specifications as shown in **Table.30**.

**Table 30 Summary of rheological properties improvement with the treatment**

<b>Local bentonite sample</b>	<b>Apparent Viscosity cP</b>	<b>Plastic viscosity cP</b>	<b>Yield point lb/100 ft<sup>2</sup></b>
Ca-bentontie “not activated”	1-4	1-2	41642
Without hot/stirring Activation	6-12	2-6	3-12
Hot/stirring Activated Bentonite	18-20	3-6	12-32
Formulation 1-B of hot/stirring	30	10	40

## 6.1 Conclusions

Based on the investigations carried out and the results and discussion presented in this study the flowing conclusions can be drawn:

1. Khulays bentonite was successfully activated using 4 wt% soda ash. The suspension, swelling and viscosity improved, and it changed from calcium type to sodium type, which is the type of bentonite used in drilling fluids as viscofier and as a fluid loss control additive.
2. A novel approach of heating while stirring method was introduced to be used with soda ash activation. This method triggered the activation process and substantially changed bentonite structure.
3. Chemical characterization of Khulays bentonite verified that it is mainly montmorillonite. Simple purification methods revealed that local bentonite can be easily utilized to produce a pure bentonite with good quality.
4. Wet sieving after dispersion in water and sedimentation gave the most pure bentonite compared to mechanically dry sieving. A full purification process was proposed and described in details. It is designed from a series of grinding, sedimentation and wet sieving process.
5. The maximum viscosity achieved by soda ash addition was 12 cP, and the required viscosity by API specifications is minimum 15 cP. The proposed thermal upgrading increased this value up to 20 cP.
6. The method of combined heating and stirring with soda ash activation has increased the swelling capacity considerably. The average particle size has increased from 2.4 ~ m for untreated bentonite to 1286.9 ~ m.

7. Suspension stability was achieved, treated bentonite showed no precipitation and particles remained in dispersion state. Measuring of zeta potential for row, pure untreated and treated bentonite gave an idea about repulsive charges on the surface of the bentonite particles, moving towards more negative values with the treatment ensured that particles will remain dispersed.
8. Investigating the basic rheology of bentonite suspension showed that bentonite concentration of 6wt% can endure more bentonite weight which resulted in an increased viscosity.
9. Filtration through API filter press reflected water hold capacity of bentonite suspension. Water control improved and filtration decreased with sodium activation and with heating/stirring treatment to 28 ml which is 65 % reduction of the untreated sample, though it's still high value.
10. Heating/stirring time dependent was investigated to get the minimum heating/stirring time for a complete treatment and maximum swelling, for 1, 3, 6, 12 and 24 hrs. A minimum of 3 hrs was required to get the maximum viscosity.
11. Particles size analysis was successfully and efficiently used as an indicator to the swelling process after sodium activation and heating treatment. It was directly related to the amount of soda ash and heating/stirring time.
12. Drilling fluid formulation using the treated bentonite in a typical water based mud gave an identical rheological behavior at both low pressure/low temperature and high pressure/high temperature condition. Filtration remained at high values compared to the Mud formulated by the standard bentonite.

## 6.2 Recommendations

Based on this study the following recommendations are suggested:

1. The local bentonite is rich montmorillonite clay and should be purified and treated using the simple and cheap process described here, so as to be useable in oil well drilling application.
2. With this final product of the treatment process, the local bentonite can be effectively used as drilling fluid additives to drill the upper sections of oil well, where oil zone is not reached and formation damages caused by filtration is not critical. This will save a huge amount of the drilling cost, if these local resources of cheap drilling fluid are utilized.
3. Heating/stirring treatment for 3wt% of local bentonite gave competent rheological properties to 6wt% of the standard bentonite. This indicates that local bentonite can save 50% of the amount used in drilling fluids if other requirement are met with few additives in little weight percentages.
4. Since the standard bentonite is usually extended with polymer and other ingredients and extenders, further investigations should be carried out to improve the filtration properties of the local bentonite.



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